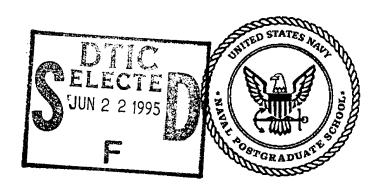
NAVAL POSTGRADUATE SCHOOL MONTEREY, CALIFORNIA



THESIS

THE ANALYSIS OF RANDOM EFFECTS REGRESSION MODEL FOR PREDICTING THE SHELF-LIFE OF GUN PROPELLANT

by

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March, 1995

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Most gun propellant is stored at depots for a long time before it is used. While being stored, the quality of the gun propellant may deteriorate and become unstable. In an attempt to avoid disaster due to use of unstable gun propellant, accurate prediction of the safe shelf-life of gun propellant is necessary. The shelf-life estimation methods used currently for a group of similar gun propellant lots are based on a fixed effects regression model. This does not take into consideration the fact that samples from the same lot are more similar than samples between lots. To capitalize on this lot-to-lot variation when estimating the shelf-life, first, a random effects regression model is developed. Secondly, a combined mixed effects model is estimated. The estimated model is then used to predict not only the shelf-life of a group of similar lots but also that of each individual lot of 5"/54 NACO gun propellant stockpile. The results indicate that, first, the claimed shelf-life is not adequate and requires amendment. Next, the group shelf-life estimated can be relatively conservative compared to the individual shelf-lives. In view of potential opportunity loss due to safe individual lots being discarded. use of individual shelf-life is recommended.

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THE ANALYSIS OF RANDOM EFFECTS REGRESSION MODEL FOR PREDICTING THE SHELF-LIFE OF GUN PROPELLANT

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ABSTRACT

Most qun propellant is stored at depots for a long time before it is used. While being stored, the quality of the gun propellant may deteriorate and become unstable. In an attempt to avoid disaster due to use of unstable gun propellant, accurate prediction of the safe shelf-life of gun propellant The shelf-life estimation methods used is necessary. currently for a group of similar gun propellant lots are based on a fixed effects regression model. This does not take into consideration the fact that samples from the same lot are more similar than samples between lots. To capitalize on this lot-to-lot variation when estimating the shelf-life, first, a random effects regression model is developed. Secondly, a combined mixed effects model is estimated. The estimated model is then used to predict not only the shelf-life of a group of similar lots but also that of each individual lot of 5"/54 NACO qun propellant stockpile. The results indicate that, first, the claimed shelf-life is not adequate and requires amendment. Next, the group shelf-life estimated can be relatively conservative compared to the individual shelf-In view of potential opportunity loss due to safe lives. individual lots being discarded, use of individual shelf-life is recommended.

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EXECUTIVE SUMMARY

Most gun propellant is stored at depots for a long time before it is used. While being stored, the quality of the gun propellant may deteriorate and become unstable. In an attempt to avoid disaster due to use of unstable gun propellant, accurate prediction of the safe shelf-life of gun propellant is necessary. Before 1914, real time storage inspection was used to estimate the safe shelf-life of gun propellant lots produced in the U.S.A. In an effort to reduce the length of time required for real time storage inspection, an Accelerated Aging Test (AAT) was introduced at Picatinny Arsenal in 1914. Under AAT, master samples taken from gun propellant lots are sequentially stored in heating chambers at 65.5 °C to measure the red nitrogen oxide fume time. Once the fume time is observed, it is related to the age of propellant stockpiles.

The current methods to estimate the shelf-life of a group of similar gun propellants are based on a fixed effects linear regression model for the fume time. The shortcomings of the current estimation methods are such that the relationship between the fume time and propellant age may not necessarily be a linear one and the fixed effects model does not take into account the potential lot-to-lot variation. To consider potential lot-to-lot variation, a random effects regression model (RERM) is required.

To understand the state-of-art estimation of the safe shelf-life of gun propellant, four technical reports are briefly reviewed. Furthermore, a preliminary analysis is conducted to understand the basic characteristics of the master samples and fleet return data sets of 5"/54 NACO gun propellant provided by Naval Surface Warfare Center (NSWC). By analyzing the fitted models and validating the model

assumptions, the standard linear regression model based on the reduced data set is selected to further investigate a random effects regression model analysis.

To consider a potential lot-to-lot variation when estimating the shelf-life, first the RERM is developed using a two-stage analysis. Next, a combined mixed effects model is estimated by employing both the maximum likelihood (ML) and restricted maximum likelihood (REML) methods. These estimators are used to define the shelf-life of the group of similar lots. The estimated model is used to predict not only the shelf-life of a group of the similar lots but also that for each individual lot by a shrinkage procedure.

The results indicate that, first, the claimed shelf-life (35 years) for the NACO propellant lots is not adequate because it overestimates the shelf-life for NACO propellant lots by almost 10 years. Therefore, it requires amendment. Next, the group shelf-life estimated can be relatively conservative compared to the individual shelf-lives. In view of potential opportunity loss due to individual lots discarded when they are still safe, use of individual shelf-life is recommended over the group shelf-life when the management of individual lots is expected to cost less than that of bulk management.

There is a nonlinear decreasing trend in fume time in some individual lots as the propellant ages. This raises questions about the approaches that use traditional linear least squares analysis of fume time data to predict safe shelf-life. This thesis recommends that a nonlinear regression analysis approach be conducted for some individual propellant lots.

I. INTRODUCTION

A. BACKGROUND

Most gun ammunition is stored at depots for a long time before being used. In order to avoid potential disaster due to storing unstable gun propellant, the shelf-life of a gun propellant stockpile should be accurately estimated. Typically, the shelf-life of gun propellant stockpile is defined as the age at which 5% of the gun propellant is unstable. The estimated shelf-life can also be used to efficiently minimize the ammunition management costs for distribution, maintenance, consumption and disposal.

In the early 1900's, real time storage inspection was used to estimate the safe shelf-life of gun propellant lots produced in the U.S.A. However, under the real time storage inspection, it took a long time to find visible changes in stability of propellent. In an effort to reduce the length of time required for the real time storage inspection, an Accelerated Aging Test (AAT) was introduced at Picatinny Arsenal in 1914 [Ref.3]. For AAT, master samples are collected for testing purposes from each gun propellant lot produced in the U.S.A. over the past sixty years. Each master sample consists of 5 pounds of propellant from each lot.

Under the AAT, forty-five grams of propellant are taken from each master sample and are stored in heating chambers at 65.5 °C until red nitrogen oxide fumes appear. The time it takes to observe such oxide fumes is called the fume time. This fume time is recorded with the corresponding propellant age. If the fume time falls below thirty days, the propellant is considered unsafe and the lot is recommended to be condemned. A fresh batch of propellant is then taken from ambient storage and the AAT is repeated. In general, it is assumed that fume time decreases as propellants age. In order to validate the results obtained from the master samples, the

AAT is employed on the fleet-return data of gun propellant lots stored in the active depots.

The classical methods used to estimate the shelf-life of a group of similar gun propellants is based on a fixed effects linear regression model. The fume time data are aggregated over a group of similar lots and a linear model for fume time is fitted against propellant age. The safe shelf-life of current methods is then estimated as the time period at which a 95% one sided lower prediction limit for the fume time curve intersects the acceptable lower specification level of thirty days of fume time. The shortcomings of the current estimation method are such that the relationship between the fume time and propellant age may not necessarily be a linear one and the fixed effects model does not take into account the potential lot-to-lot variation. Other relationships in addition to the linear model should be examined. In order to consider potential lot-to-lot variations, a random effects model is required.

To understand the state-of-art related to the estimation of the safe shelf-life of gun propellant, four technical reports are briefly reviewed.

B. LITERATURE REVIEW

In the report entitled "Prediction of Safe Life of Propellants" [Ref. 1], the nature of 65.5°C surveillance test and the propellant chemical deterioration are briefly discussed. In this study, it is recognized that the measurement of residual stabilizer content offers the best means of establishing the stability potential of propellant. This report also discusses the results obtained from the standard artillery propellant when exposed to the aging test at various temperatures.

The measurements of residual stabilizer content versus time were used as the proxy for propellant deterioration. For

interpretation of this deterioration phenomena, Berthelot's Law was employed. It demonstrated that a family of straight lines can be plotted characterizing the length of time necessary at the various test temperatures to obtain a given variation of stabilizer content. Finally, by establishing realistic cut-off points regarding stabilizer content in a given storage temperature, propellant safe life was estimated.

Secondly, the report entitled "Type Life Program History, Philosophy, Accomplishment, And Future" [Ref. 2] introduces the concept of the type life test. The type life test is derived from the AAT approach and takes into account a more realistic temperature. In this study, accelerated aging techniques are used to observe the aging mechanisms and characteristics of propulsion ordnance along with associated components. They are also use to determine how age affects the performance of the units.

The type life temperature-time profile defined in this study is based on the "hot-month" concept, and differs from the AAT and its use of constant testing temperature. The concept is briefly stated as follows: (1) propellants are a chemical system; (2) chemical systems deteriorate more rapidly at elevated temperatures; and (3) in a normal year's time, the deterioration occurs only during the summer months.

Based on "hot-month" concept, the engineers establish a temperature-time profile termed the "compressed-ambient cycle" and consisting of 26 weeks. This profile was designed to simulate the four seasons of the year and is related to the aging that normally occurs during magazine storage in the U.S.A. There is a 16-week period at 100 °F (38 °C) representing a long severe summer time, two 3-week periods at 70 °F representing spring and autumn, and a 4-week period at 40 °F representing winter. The compressed-ambient cycle (26 weeks or 1/2 year) simulates one year of magazine storage; therefore there is a 2:1 (i.e.52 weeks:26 weeks) aging ratio. The main

purpose of this report was to evaluate the effect of the loss of nitroglycerin from the propellant into the inhibitor. This loss results in propellant cracking and/or a reduction in propulsion performance due to a loss of energy.

Thirdly, a report entitled "Statistical Analysis of ARDEC'S Fume Data Base" [Ref. 3] describes the procedure for AAT based on the master sample surveillance program in detail. Three types of propellants were used separately based on single and double base propellants. The single and double bases are terms of ammunition, distinguished by the methods of ignition. The single base propellant can be ignited by either mechanical or electrical means. The double base propellant can be ignited using both methods. In this study, fume times are plotted against sampling times for each propellant form. Not only is the first order linear relationship examined with this approach but also other relationships such as polynomial regressions, logarithmic and exponential transformation are considered to establish fume time behavior over the life of the propellant.

Finally, a study entitled "Long-Term Stability of Navy Gun Propellants" [Ref. 4] was initiated to standardize the requirements of safety surveillance testing between the Army was necessary because current Navy Navy. This surveillance testing includes a 65.5 °C oven fume test (AAT) on single-base propellants and double-base propellants with 10% nitroglycerin content. or equal to less t.han Additionally, propellants must take more than 30 days to fume to be considered stable. In contrast, the Army's disposition criteria is based on a stabilizer analysis of less than 0.2% nitroglycerin content. Because a substantial quantity of Navy propellant is being currently stored in Army depots, nonstandardization complications commonly occur. This study was tasked by Naval Sea Systems Command(NSSC) to determine the critical level of propellant stabilizer for

propellants of interest to the Navy and to the Marine Corps, to develop the best test methods to evaluate that level of criticality for Navy propellants, and to apply the data for standardization.

The test was conducted on propellants removed from loaded ammunition. Each sample was subjected to chemical, kinetic, and physical properties analyses. As a result of these analyses, it was recommended to continue use of the 65.5 °C oven fume test because it is the procedure that provides the most complete coverage of propellant in storage worldwide. The Navy has a large historical database of fume data, and statistical analysis of the data provides an inexpensive, comprehensive means of insuring that Navy-developed propellant is safe for worldwide storage.

In summary, the master sample surveillance test is recommended for continuous use to determine safety of the gun propellant in storage. It is proposed that the relationship between the fume time and propellant age may not be limited to a linear relationship. The management of data structure and the data transformation (e.g. logarithmic or reciprocal) is required. Additionally, no potential lot-to-lot variation in shelf-life estimation was examined in the four technical reports summarized here.

C. THE SCOPE OF THESIS

In this thesis, the master sample surveillance test data set is first examined using several fixed effects regression models in an attempt to find the relationship between the fume time and propellant age. Then, a random effects regression model is introduced to take into account a lot-to-lot variation in fitting the relationship between the fume time and propellant age. Finally, results are discussed and recommendations are made.

II. FIXED EFFECTS REGRESSION MODEL

A. DATA DESCRIPTION

A preliminary analysis is conducted to understand the basic characteristics of the master sample data set provided by Naval Surface Warfare Center (NSWC). The data set was collected based on the oven test data consisting of 28 lots of master samples of 5"/54 NACO gun propellant. They were manufactured at Badger Army Ammunition Plant (BAAP), Baraboo, Wisconsin between 1972 and 1974. The NACO is a generic term of NAvy COol representing a family of cool-burning, single-base propellants. These propellants are made from low-nitration nitrocellulose with a coolant, a stabilizer, a decoppering agent, and a flash suppressant added.

First by subtracting the manufacturing date (see Appendix A) from the date when a 45q propellant batch was sent to the heating chamber, the age is computed for each propellant lot. The data of the fume time and propellant age is shown in computer program list in Appendix B. Both descriptive statistics of fume time and propellant age are displayed in In summary, 221 observations of fume time and Table 1. propellant age had been taken from 28 propellant lots. maximum (1492 days) and minimum (19 days) of fume time are observed during 6813 days of propellant age. attempt to find the relationship between the fume time and propellant age, the fume time is plotted against propellant age in Figure C.1. It is observed that there is an unexpected increasing trend in fume time over propellant age up to 1100 days. This unexpected pattern may be due to the type of ammunition, shape of propellant container or caused by some unknown chemical reaction. [Ref. 3]

Des	criptive Statis	stics For Raw Data						
manufact	ure date	min 1972 max 1974						
number	of lot	28						
number of measur	repeated ements	221						
	mean	802.61						
fume time in days	std	222.159						
in days	max	1492						
	min	19						
	mean	3488.824						
propellant age in days	std	2020.824						
age in days	max	6813						
	min	0						

Table 1. Descriptive Statistics For Raw Data

B. STANDARD LINEAR REGRESSION MODEL I

Regardless of such unexpected pattern, according to the current practice, standard linear regression model I, is applied to fit the fume time (Y) against propellant age (X):

$$Y = B_0 + B_1 X + \epsilon$$
 (1)

where B_0 and B_1 represent the expected initial condition and deterioration rate of stability of gun propellant, and ε is the random error which follows a Normal (0, τ^2) distribution.

The predicted value (\tilde{Y}_*) at X=X $_*$ is

$$\tilde{Y}_* = \hat{B}_0 + \hat{B}_1 X_*$$
 (2)

where \hat{B}_0 and \hat{B}_1 are the ordinary least square estimators of B_0 and B_1 . Subsequently a 90% prediction interval for a fume time at given propellant age (X_*) is

$$\tilde{Y}_* \pm t \ (0.95, N-2) SEPRED \ (\tilde{Y}_* \mid X_*),$$
 (3)

where N is the total number of observations, t(0.95,N-2) is the 95th percentile of the t distribution with N-2 degrees of freedom. SEPRED $(\tilde{Y}_* \mid X_*)$ is the standard error of predicted \hat{Y}_* at X=X*:

SEPRED
$$(\tilde{Y}_* \mid X_*) = \hat{\tau} \left[1 + \frac{1}{N} + \frac{(X_* - \overline{X})^2}{\sum (X_* - \overline{X})^2}\right]^{\frac{1}{2}}.$$
 (4)

Based on this information, the group shelf-life (t_{sl}) can be estimated by checking the time period when the 95% one-sided lower prediction limit intersects 30 days of fume time:

$$30 = \hat{B}_0 + \hat{B}_1 t_{sl} - t (0.95, N-2) SEPRED (\tilde{Y}_* | t_{sl}).$$
 (5)

Next, actual data obtained from the NACO propellant surveillance test is used to fit the model and to estimate the shelf-life. The fitted model based on (2) is given in Table

Standard Linear R	egression Model I
$\hat{\mathcal{B}}_0$	1045.510
$\hat{\mathcal{B}}_{\mathtt{l}}$	-0.070
$SE(\hat{B}_0)$	23.171
$SE(\hat{B_1})$	0.006
R-square	0.4009
lack of fit test	degrees of freedom: 200,19; P value = 0
Normal distribution test (Kolmogorov-Smirnov Test)	P value = 0.004 not Normal
estimated shelf-life	10400 days (28.49 years)

Table 2. Fitted Standard Linear Regression Model I

2 and displayed as a solid line in Figure C.1. A 90% two-sided prediction interval (solid curves) is overlaid in Figure C.1. The resultant shelf-life based on (5) turns out to be 10400 days (28.49 years) as shown in Figure C.2.

The residual analysis $(r_i = Y_i - \hat{Y}_i$, for i = 1...N) for standard linear regression model I is shown in Figure C.3. Residual pattern indicates nonlinearity and nonconstant variance. Also, the Kolmogorov-Smirnov test statistic shown in Table 2 indicates that the residuals do not follow a normal distribution.

For further data analysis, the data set that covers from zero to 1100 days of propellant age is deleted, because the pattern is unstable during this early stage and this pattern

is not expected to last longer than 1100 days. Furthermore, after 1100 days of propellant age, the fume time tends to decrease as propellants age and this pattern is more Therefore, the use of the remaining data set consistent. appears to be reasonable for the shelf-life estimation. following three regression models are employed (1) standard regression model ΙI (delete observations correspond to propellant age below 1100 days and use standard linear regression model I);(2) linearizing transformation model I (transform propellant age to 1/ propellant age); and (3) linearizing transformation model II (transform fume time to log(fume time)). Graphical method precedes statistical analysis.

C. STANDARD LINEAR REGRESSION MODEL II

The standard linear regression model II is essentially the same as the standard linear regression model I(1). However, parameters are estimated based on a reduced data set. After deleting the early age, the fume time is plotted against propellant age in Figure C.4. The results of model fit are shown in Table 3. The predictive value (middle solid line) and a 90% two-sided prediction interval for a fume time (solid curves) are overlaid in Figure C.4.

The residual analysis for the standard linear regression model II is shown in Figure C.5. In general, the plot of residuals against fitted values over fume time between zero day and 750 days (propellant age above 4500 days) does not show any systematic features and they appear to have a common variance. But, a minor nonrandom pattern is observed when the fume time exceeds 750 days (propellant age below 4500 days). Also, based on the p-value associated with Kolmogorov-Smirnov test shown in Table 3, we can conclude the residual of this model follows a normal distribution.

Standard Linear Re	egression Model II
$\hat{\mathcal{B}}_{0}$	1388.452
$\hat{\mathcal{B}}_1$	-0.141
$SE(\hat{B}_0)$	20.612
$SE(\hat{B_1})$	0.005
R-square	0.838
lack of fit test	degrees of freedom: 167,11; P value = 0.001
Normal distribution test (Kolmogorov-Smirnov Test)	P value = 0.458 Normal
estimated shelf-life	8469 days (23.20 years)

Table 3. Fitted Standard Linear Regression Model II

By checking the level of 95% one-sided lower prediction limit for a fume time that intersects 30 days of fume time in Figure C.6, the estimated group shelf-life turns out to be 8469 days (23.20 years).

D. LINEARIZING TRANSFORMATION MODEL I

In linearizing transformation model I, it is assumed that the fume time is linearly related to reciprocal of propellant age:

$$Y = B_0 + B_1 \left(\frac{1}{X} \right) + \epsilon$$
 (6)

The fume time is plotted against propellant age in Figure C.7.

The results of a model fit are shown in Table 4. The predicted value for the fume time $\tilde{Y}_* = \hat{B}_0 + \hat{B}_1 \; (\frac{1}{X_*})$ is shown

Linearizing Trans	formation model I
$\hat{\mathcal{B}}_0$	411.738
$\hat{\mathcal{B}}_1$	1315827.380
$SE(\hat{B_0})$	17.420
$SE(\hat{\mathcal{B}}_1)$	51308.706
R-square	0.787
lack of fit test	degrees of freedom: 167,11; P value = 0
Normal distribution test (Kolmogorov-Smirnov Test)	P value = 0.02948 not Normal
estimated shelf-life	∞

Table 4. Fitted Linearizing Transformation Model I

as a middle curve and a 90% two-sided prediction interval for a fume time are overlaid in Figure C.7.

It is interesting to note that the 95% one-sided lower prediction limit for predictive fume time converges to around 400 days as the propellant age continuously increases. Therefore, the safe shelf-life of gun propellant based on linearizing transformation model I cannot be estimated or can be considered as infinity.

The residual analysis for the linearizing transformation model I is shown in Figure C.8. In general, the plot of

residuals against fitted values over fume time between 400 days and 800 days (propellant age above 4000 days) does not show any systematic features and they appear to have a common variance. But a nonrandom pattern is observed when the fume time exceeds 800 days (propellant age below 4000 days). This phenomenon is similar to the standard linear regression model II (see Figure C.5). Also, based on the p-value associated with Kolmogorov-Smirnov test shown in Table 4, we can conclude that the residual of this model does not follow a normal distribution.

E. LINEARIZING TRANSFORMATION MODEL II

In linearizing transformation model II, it is assumed that the propellant age is linearly related to log fume time:

$$LOG (Y) = B_0 + B_1 X + \epsilon.$$
 (7)

The fume time is plotted against propellant age in Figure C.9. The results of a model fit are shown in Table 5. The predicted value for the fume time $\tilde{Y}_* = EXP \ (\hat{B}_0 + \hat{B}_1 X_*)$ and a 90% two-sided prediction interval for a fume time are overlaid in Figure C.9.

The residual analysis for the linearizing transformation model II is shown in Figure C.10. In general, the plot of residuals against fitted values over fume time between zero day and 6.6 days (propellant age above 4000 days) does not show any systematic features and they appear to have a common variance. But a nonrandom pattern is observed when the fume time exceeds 6.6 days (propellant age below 4000 days). This phenomenon is also similar to the previous two models (see Figure C.5 and Figure C.8). Also, based on the p-value associated with Kolmogorov-Smirnov test shown in Table 5, we

Linearizing Trans	formation model II
$\hat{\mathcal{B}}_{0}$	7.38
\hat{B}_1	-1.79 E-4
$SE(\hat{B}_0)$	5.75 E-2
$SE(\hat{B_1})$	1.29 E-5
R-square	0.520
lack of fit test	degrees of freedom: 167,11; P value = 0
Normal distribution test (Kolmogorov-Smirnov Test)	P value = 1.0E-11 not Normal
estimated shelf-life	17400 days (47.67 years)

Table 5. Fitted Linearizing Transformation Model II

can conclude that the residual of this model does not follow a normal distribution.

By checking the level of the 95% one-sided lower prediction limit of a fume time that intersects 30 days of fume time in Figure C.11, the estimated group shelf-life turns out to be 17400 days (47.67 years).

In summary, by analyzing the fitted models and validating the model assumptions of the various fixed effects regression models, the effective distribution data model appears to be the standard linear regression model II based on the reduced data. This model is compared to the model fit based on the fleet return data.

F. STANDARD REGRESSION MODEL BASED ON FLEET RETURN DATA

This model is based on the oven test data consisting of 28 lots of fleet return data of 5"/54 NACO gun propellant. The descriptive statistics of fume time and propellant age are displayed in Table 6. In summary, 204 observations of fume

	Descriptive	Statistics							
manufact	ure date	min 1972 max 1974							
number	of lot	28							
number of	repeated	202							
measur	ements								
	mean	702.12							
fume time in days	std	85.46							
111 00,2	max	993							
	min	479							
	mean	2822							
propellant age in days	std	1388.8							
	max	5074.5							
	min	361							

Table 6. Descriptive Statistics For Fleet Return Data

time and propellant age had been taken from 28 propellant lots. The maximum (993 days) and minimum (479 days) of fume time are observed during 5074.5 days of propellant age. The fume time is plotted against propellant age in Figure C.12. The results of model fit which is based on standard linear

Standard Linear B	Regression Model
$\hat{\mathcal{B}}_{0}$	823.375
$\hat{\mathcal{B}}_1$	-0.042
$SE(\hat{B_0})$	9.793
$SE(\hat{\mathcal{B}}_1)$	0.003
R-square	0.422
lack of fit test	degrees of freedom: 127,73; P value = 0.0000
Normal distribution test (Kolmogorov-Smirnov)	P value = 0.0040 not Normal
estimated shelf-life	15700 days (43 years)

Table 7. Fitted standard Linear Regression Model
Based on Fleet Return Data

regression model I are shown in Table 7. The predicted value (middle solid line) and a 90% prediction interval for a fume time (solid curves) are overlaid in Figure C.12. The resultant shelf-life based on (5) of this model turns out to be 15700 days (43 years) as shown in Figure C.13.

The residual analysis for this model is shown in Figure C.14. There is a right-opening megaphone pattern in Figure C.14. This residual pattern observed shows nonconstant variance. Also, the Kolmogorov-Smirnov test statistic shown in Table 7, indicates that the residuals of this model do not follow a normal distribution. Two remedies for nonconstant variance (Weisberg [Ref. 8]) are made using weighted least square estimation and power transformation:

1. Weighted Least Square Model

The first remedy is the weighted least square model. The variance of the random error in the standard linear regression model I (1) appears to increase as X increases, that is, τ =KX where K is a positive constant. Therefore, the model (1) is modified as follows:

$$\frac{Y}{X} = \frac{B_0}{X} + B_1 + \frac{\epsilon}{X}$$
 (8)

The residual analysis for this model is shown in Figure C.15. It still shows a right-opening megaphone pattern in Figure C.15 and the suggested weighted least square method is not adequate.

2. Power Transformation Model

The second remedy is to transform the response Y via a variance stabilizing transformation. By checking the residual pattern of several transformation models, the following transformation model appears to satisfy the assumption of common variance (see Figure C.16):

$$Y^{-3} = B_0 + B_1 X + \epsilon. {9}$$

The fume time is plotted against propellant age in Figure C.17. The results of a model fit are shown in Table 8. The prediction fume time $\tilde{Y} = (\hat{B}_0 + \hat{B}_1 X_*)^{-\frac{1}{3}}$ and a 90% prediction interval for a fume time are overlaid in Figure C.17.

The p-value associated with Kolmogorov-Smirnov test of residual of model (9) indicates that the residual of this model follows a normal distribution.

Power Transformation model	
\hat{B}_0	1.75 E-9
$\hat{\mathcal{B}}_1$	4.87 E-13
$SE(\hat{B}_0)$	1.26 E-10
$SE(\hat{B_1})$	4 E-14
R-square	0.425
lack of fit test	degrees of freedom: 127,73; P value = 0.001
Normal distribution test (Kolmogorov-Smirnov)	P value = 0.18964 Normal
estimated shelf-life	∞

Table 8. Fitted Power Transformation Model

The shelf-life is estimated based on the following formula:

$$30 = [\hat{B}_0 + \hat{B}_1 t_{sl} - t(0.95, N-2)]^{-\frac{1}{3}}.$$
 (10)

The estimated group shelf-life turns out to be infinite.

This shelf-life estimation based on fixed effects regression models does not take into account the lot-to-lot variation. Apparently potential lot-to-lot variation is supported by individually fitted models based on the Standard Linear Regression Model I, II and fleet return data as shown

in Figures C.18, C.19, C.20. Additionally, this variation can be seen by checking linearizing transformation model I after smoothing the data following Lowess procedure [Ref. 9] as shown in Figure C.21.

In the next chapter, standard linear regression model II will be further investigated for a random effects regression model analysis.

III. RANDOM EFFECTS REGRESSION MODEL

A. INTRODUCTION

The current methods used to estimate the shelf-life of qun propellant is based on the standard linear regression In this fixed effects linear regression model, the expected initial fume time (B_0) and deterioration rate (B_1) are assumed to be constant for all gun propellant lots. However, in practice, the performance varies among lots even though a group of lots is manufactured based on the same technique. This kind of phenomenon can be observed in the individually fitted models in Figure C.18 to C.21. The lot-to-lot variation is observed not only in the intercepts (expected initial condition) but also in slopes (deterioration rate) among a group of similar lots. The current methods does not take into account this kind of constant. In order to consider potential lot-to-lot variation within the group, a random (or mixed effects) regression model effects (RERM) necessary.

In this chapter, first, the RERM will be introduced based on a two-stage analysis. Next, a combined two-stage model (mixed effects model) will be fitted based on the reduced data of the master samples provided by NSWC. In order to obtain the estimators of unknown parameters in mixed effects model, the maximum likelihood estimation (MLE) and restricted maximum likelihood (REML) will be employed. These estimators are, then, used to defined the shelf-life of the group of similar lots that takes into account the lot-to-lot variation. Furthermore, a shrinkage procedure will also be introduced to estimate the shelf-life of an individual lot.

B. MODEL

1. Within-Lot Model

In order to estimate the deteriorating patterns of gun propellant lots over storage time, each 45g batch is taken from 5 pound master sample of lot i and its ageing is accelerated at the heating chamber at 65.5 ^{0}C . The fume time (y_{ij}) of lot i (i=1...N) is recorded with the corresponding propellant age (t_{ij}) (j=1...n_i) whenever the red nitrogen oxide fume appears. Once repeated measurements are obtained, we attempt to find the relationship between the fume time and propellant age. The following within-lot model is used to describe the relationship between the fume time and the age of a lot:

For i=1...N and $j=1...n_i$

$$y_{ij} = \alpha_{0i} + \alpha_{1i} t_{ij} + \epsilon_{ij}$$
 (11)

where α_{0i} and α_{1i} represents the expected initial condition and deterioration rate of the stability of propellant lot i, and ϵ_{ij} is the random error which is assumed to follow a independent N(0, τ^2).

2. Between-Lot Model

In order to take into account the lot-to-lot variation among intercepts (expected initial condition) and slopes (deterioration rate) of a group of lots, we assume the following between-lot model:

$$\alpha_{0i} = w_i \gamma_0 + \delta_{0i}$$

$$\alpha_{1i} = w_i \gamma_1 + \delta_{1i}$$
(12)

where w_i is a 1 \times k vector representing values of K factors that may cause varying α_{0i} and α_{1i} . The γ_0 and γ_1 are k \times 1 vectors of regression coefficients for α_{0i} and α_{1i} , respectively. The δ_{0i} and δ_{1i} are assumed to follow independent

$$N(0,\Sigma)$$
, where $\Sigma = \begin{bmatrix} \sigma_0^2 & \sigma_{01}^2 \\ \sigma_{01}^2 & \sigma_1^2 \end{bmatrix}$. Note that for AAT, the master

samples are put in the constant temperature condition (65.5 ^{0}C) and w_{i} in this case is 1.

When α_{0i} and α_{1i} in (11) are substituted with those in (12), a combined mixed effects model and is formulated as follows:

For i=1...N and $j=1...n_i$,

$$y_{ij} = (w_i \gamma_0 + \delta_{0i}) + (w_i \gamma_1 + \delta_{1i}) t_{ij} + \epsilon_{ij}$$

$$= (w_i \gamma_0 + w_i \gamma_1 t_{ij}) + (\delta_{0i} + \delta_{1i} t_{ij}) + \epsilon_{ij}$$

$$= (w_i, w_i t_{ij}) \left(\frac{\gamma_o}{\gamma_1}\right) + (1, t_{ij}) \left(\frac{\delta_{oi}}{\delta_{1i}}\right) + \epsilon_{ij} .$$
(13)

This mixed effects model can be re-written using matrix notation:

$$y = X\beta + Z\nu + e , \qquad (14)$$

where y is a $n_i \times 1$ vector of y_{ij} ; β is a 2K×1 coefficient vector $(\gamma_0$, $\gamma_1)'$ associated with a $\sum_{i=1}^N n_i \times 2K$ matrix of X consisting of

 $(w_i$, $w_i t_{ij})$; v is a $2n_i \times 1$ vector of $(\delta_{0i}$, $\delta_{1i})'$ associated with a known $\sum_{i=1}^N n_i \times 2n_i$ block diagonal matrix Z of (1 , t_{ij}), and e

is an $\sum_{i=1}^{N} n_i \times 1$ vector of random error. Assume that v and e

are uncorrelated and have expections 0 and variances G and R, respectively, where both G and R are nonsingular. applying these assumptions to (14) leads to

$$E(y) = X\beta$$

$$Var(y) = V = ZGZ' + R,$$
(15)

where G is a $2n_i \times 2n_i$ block diagonal matrix consisting of $\begin{bmatrix} \sigma_0^2 & \sigma_{01}^2 \\ \sigma_{01}^2 & \sigma_1^2 \end{bmatrix} \text{ and } R = Var(e) = \tau^2 I_N \sum_{i=1}^{N} n_i.$

When G and R (and hence V) are known, estimates of both β and the realized value of v are

$$\hat{\beta} = [\hat{\gamma}_{0}, \hat{\gamma}_{1}]' = (X'V^{-1}X)^{-1}X'V^{-1}y$$

$$\hat{v} = [\hat{v}_{1}, \dots \hat{v}_{N}]' = [\hat{\delta}_{01}, \hat{\delta}_{11}, \dots \hat{\delta}_{0N}, \hat{\delta}_{1N}]' = GZ'V^{-1}(y - X\hat{\beta})$$
(16)

respectively, where β is the best linear unbiased estimator (BLUE) of β , and $\hat{\mathbf{v}}$ is the best linear unbiased predictor (BLUP) of v. They can also be obtained from Henderson's (1984) mixed model equation [Ref. 10]:

$$\begin{bmatrix} X'R^{-1}X & X'R^{-1}Z \\ Z'R^{-1}X & Z'R^{-1}Z+G^{-1} \end{bmatrix} \begin{bmatrix} \mathbf{\hat{\beta}} \\ \hat{\mathbf{v}} \end{bmatrix} = \begin{bmatrix} X'R^{-1}y \\ Z'R^{-1}y \end{bmatrix}$$
(17)

where $\boldsymbol{\beta} = [\hat{\gamma}_0, \hat{\gamma}_1]'$ and $\hat{\mathbf{v}} = [\hat{\boldsymbol{\delta}}_{01}, \hat{\boldsymbol{\delta}}_{11}, \dots \hat{\boldsymbol{\delta}}_{0N}, \hat{\boldsymbol{\delta}}_{1N}]'$. When G and R (and hence V) are unknown, the estimator of V can be obtained by maximizing the logarithm of likelihood function [Ref. 10]

$$\log L = -\frac{1}{2} N \log_2 \pi - \frac{1}{2} \log |v| - \frac{1}{2} (y - X\beta) / V^{-1} (y - x\beta).$$
 (18)

The ML estimators \hat{V} and $\hat{oldsymbol{\beta}}$ can be obtained by solving the following equations simultaneously

$$X' \hat{V}^{-1} X \hat{B} = X' \hat{V}^{-1} V$$
 (19)

and

$$tr(\hat{V}^{-1}Z_{i}Z_{i}') = (y-X\hat{\beta})'\hat{V}^{-1}Z_{i}Z_{i}'\hat{V}^{-1}(y-X\hat{\beta}) \qquad for \ i=0,1,...N,$$
 (20)

where Z_i is a $\sum_{i=1}^N n_i \times 2n_i$ block diagonal matrix of $(1,t_{ij})$ and

$$tr(\hat{V}^{\text{-1}}Z_iZ_i') \text{ is trace of the } \sum_{i=1}^N n_i \times \sum_{i=1}^N n_i \text{ matrix } (\hat{V}^{\text{-1}}Z_iZ_i') \text{ that } i = 1, \dots, n_i$$

denotes the sum of the diagonal entries. An algebraically simpler expression for (20) is derived by defining

$$P = V^{-1} - V^{-1}X(X'V^{-1}X)^{-1}X'V^{-1}.$$
 (21)

Then from (19) it is clear that for \hat{P} being P with V replaced by \hat{V}

$$\hat{V}^{-1}(y - X\hat{\beta}) = \hat{P}y, \tag{22}$$

so that the ML estimators of \hat{V} and $\hat{m{\beta}}$ also can be re-written by solving the following ML equations simultaneously

$$X' \hat{V}^{-1} X \hat{\beta} = X' \hat{V}^{-1} Y$$
 (23)

and

$$\left\{_{c} tr(\hat{V}^{-1}Z_{i}Z_{i}')\right\}_{i=0}^{N} = \left\{_{c} y'\hat{P}Z_{i}Z_{i}'\hat{P}y\right\}_{i=0}^{N},$$
 (24)

where $\{_c \ tr \ (\hat{V}^{-1}Z_iZ_i') \ \}_{i=0}^N$ is $(N+1)\times 1$ column vector with elements which are the trace of the matrix $(\hat{V}^{-1}Z_iZ_i')$, for $i=0,1,\ldots N$, respectively.

Also, the estimator of V can be obtained by restricted maximum likelihood (REML). A basic idea of REML estimation is that of estimating variance components based on residuals calculated after fitting by ordinary least squares just the fixed effects part of the model. The REML estimator can also by viewed as maximizing a marginal likelihood. The REML equations can therefore be derived from the ML equations of (24). By making suitable replacements, the REML estimator \hat{V}

can be obtained by solving the following equation:

$$\{ tr[(K'\hat{V}K)^{-1}K'ZZ'K] \}_{i=0}^{r} =$$

$$\{ y'K(K'\hat{V}K)^{-1}K'ZZ'K(K'\hat{V}K)^{-1}K'y \}_{i=0}^{r}$$
(25)

with $P = K(K'VK)^{-1}K'$ where K'X=0.

When the resultant \hat{V} is found by solving the ML equations ((19),(20)) or REML equation (25), the Henderson's mixed model equation can be re-written as follows:

$$\begin{bmatrix} X'\hat{R}^{-1}X & X'\hat{R}^{-1}Z \\ Z'\hat{R}^{-1}X & Z'\hat{R}^{-1}Z + \hat{G}^{-1} \end{bmatrix} \begin{bmatrix} \hat{\beta} \\ \hat{\mathbf{v}} \end{bmatrix} = \begin{bmatrix} X'\hat{R}^{-1}Y \\ Z'\hat{R}^{-1}Y \end{bmatrix} . \tag{26}$$

Finally, $\hat{\boldsymbol{\beta}} = \left[\hat{\boldsymbol{\gamma}}_0 \ , \ \hat{\boldsymbol{\gamma}}_1\right]'$ and $\hat{\boldsymbol{\nu}} = \left[\hat{\boldsymbol{\delta}}_{01}, \hat{\boldsymbol{\delta}}_{11}, \ldots \hat{\boldsymbol{\delta}}_{0N}, \hat{\boldsymbol{\delta}}_{1N}\right]'$ substitutes $\boldsymbol{\beta} = \left[\boldsymbol{\gamma}_0 \ , \boldsymbol{\gamma}_1\right]'$ and $\boldsymbol{\nu} = \left[\boldsymbol{\delta}_{01}, \hat{\boldsymbol{\delta}}_{11}, \ldots \hat{\boldsymbol{\delta}}_{0N}, \hat{\boldsymbol{\delta}}_{1N}\right]'$ in (14) as follows:

$$\hat{y} = X \hat{\beta} + Z \hat{v} . \tag{27}$$

In fume time analysis, the test temperature condition is constant and W_i in this analysis is 1. Therefore, $X_{ij} = (W_i, W_i t_{ij}) = (1, t_{ij})$ and $Z_{ij} = (1, t_{ij})$. Then (27) becomes

$$\hat{y} = X\hat{\beta} + Z\hat{v} = Z\hat{\beta} + Z\hat{v}$$

$$= Z\hat{\beta} + Z(\hat{G}Z'\hat{V}^{-1}(y - Z\hat{\beta}))$$

$$= Z\hat{\beta} + Z\hat{G}Z'\hat{V}^{-1}y - Z\hat{G}Z'\hat{V}^{-1}Z\hat{\beta}$$

$$= (I - Z\hat{G}Z'\hat{V}^{-1})Z\hat{\beta} + (Z\hat{G}Z'\hat{V}^{-1})y$$

$$= (I - H)\hat{E}(Y) + HY$$

$$(28)$$

where $H=Z\hat{G}Z'\hat{V}^{-1}$ is the weighted of mean of $\hat{E}[y]$ and y. The resulting \hat{Y} is called a shrinkage estimator.

The predicted value of fume time (\tilde{y}_{ij}) of lot i at t_{ij} can be estimated as

$$\tilde{y}_{ij} = x_{ij} \begin{pmatrix} \hat{\gamma}_0 \\ \hat{\gamma}_1 \end{pmatrix} + z_{ij} \begin{pmatrix} \hat{\delta}_{0i} \\ \hat{\delta}_{1i} \end{pmatrix} = (1, t_{ij}) \begin{pmatrix} \hat{\gamma}_0 \\ \hat{\gamma}_1 \end{pmatrix} + (1, t_{ij}) \begin{pmatrix} \hat{\delta}_{0i} \\ \hat{\delta}_{1i} \end{pmatrix}. \tag{29}$$

A 90% prediction interval for a fume time at given propellant age t_{ij} is

$$\tilde{y}_{ij} \pm t \text{ (0.95 , N-2) } SEPRED \text{ (} \tilde{y}_{ij} \mid t_{ij} \text{)}$$

where

$$SEPRED(\tilde{y}_{ij}|t_{ij}) = [(1,t_{ij}) \hat{V}(\hat{\beta}_i) (1,t_{ij})' + (1,t_{ij}) \hat{V}(\hat{v}_i) (1,t_{ij})' + \hat{\tau}^2]^{\frac{1}{2}};$$
(31)

and $\boldsymbol{\hat{V}}$ $(\boldsymbol{\hat{\beta}}_{i})$ and $\boldsymbol{\hat{V}}$ $(\boldsymbol{\hat{v}}_{i})$ is the ith block diagonal matrices of

$$\hat{V}(\hat{\beta}) = (X' \hat{V}^{-1} X)^{-1};$$

$$\hat{V}(\hat{v}) = \hat{G} Z' \hat{V}^{-1} \hat{G} Z'.$$
(32)

In order to estimate deteriorating patterns of a group of similar lots, one would be interested in the shelf-life of the group of similar lots (t_{sl}) that takes into account the lot-to-lot variation. It can be obtained as follows by only considering the group effects:

$$30 = x_{ij} \ \beta - t \ (0.95, N-2) \ SEPRED_{(group)} \ (\tilde{y} \mid t_{sl})$$

$$= (1, t_{sl}) \begin{pmatrix} \hat{\gamma}_0 \\ \hat{\gamma}_1 \end{pmatrix} - t \ (0.95, N-2) \ SEPRED_{(group)} \ (\tilde{y} \mid t_{sl})$$
(33)

where $SEPRED_{(group)}$ ($\tilde{y} \mid t_{sl}$) is the standard error of predicted \hat{y} of a group given by

$$SEPRED_{(group)} (\tilde{y}|t_{ij}) = [(1, t_{ij}) \hat{V}(\hat{\beta}_i) (1, t_{ij})^{\prime} + \hat{\tau}^2]^{\frac{1}{2}}.$$
 (34)

The group shelf-life can be compared to the individual shelf-life (t_{ssl}) based on the shrinkage estimation (28). It is defined as the time period at which the expected fume time curve intersects the 30 days of fume time:

$$30I = X\hat{\beta} + Z\hat{v}$$

$$= (I - Z\hat{G}Z'\hat{V}^{-1'})Z\hat{\beta} + Z\hat{G}Z'\hat{V}^{-1}y$$
(35)

where I is a $n_i \times 1$ vector of 1's and Z is a $n_i \times 1$ vector of $(1, t_{ssl})$.

C. DATA ANALYSIS

Standard linear regression model II based on the reduced data is further investigated for the random effects regression model analysis. As explained earlier, under the AAT, the fume time (y_{ij}) of each lot is repeatedly measured five to nine times in an accelerated condition (65.5 0 C) during the last 20 years. The age (t_{ij}) for each of the lot is computed by subtracting the manufacturing date (see Appendix A) from the date in which when a 45g propellant batch was sent to the heating chamber.

First, in order to estimate the safe shelf-life of a group of similar gun propellants based on the random effects regression model, the within-lot and between-lot models were combined to be a mixed effects model (14). Since the G and R (hence V) are unknown (15), the estimators of G and R can be obtained by a restricted maximum likelihood (REML) (25):

$$\hat{G} = V \hat{a} r(v) = \begin{bmatrix} \hat{\sigma}_0^2 & \hat{\sigma}_{01}^2 \\ \hat{\sigma}_{01}^2 & \hat{\sigma}_1^2 \end{bmatrix} = \begin{bmatrix} 0.07079100 & -0.00450653 \\ -0.00450653 & 0.00030661 \end{bmatrix}$$

and $\hat{R} = V \hat{a} r(e) = 0.05926819$. Unknown parameters β and ν can in turn be estimated by Henderson's mixed equation (26): (1) $\hat{\beta} = \left[\hat{\gamma}_0, \hat{\gamma}_1\right]' = \left[3.81021477, -0.14105394\right]' \text{ where } \operatorname{se}\left[\hat{\gamma}_0\right] = \left[0.07176290\right] \text{ and } \operatorname{se}\left[\hat{\gamma}_1\right] = \left[0.00534921\right]; (2) \hat{\mathbf{v}} = \left[\hat{\delta}_{01}, \hat{\delta}_{11}, \ldots \hat{\delta}_{0N}, \hat{\delta}_{1N}\right]'$ and the corresponding standard errors are shown in Table 9.

INTERCEPT LOTID 11142 -0.06196541 0.19329612 AGE LOTID 11142 0.00479259 0.01346718 INTERCEPT LOTID 11143 -0.11611040 0.18778713 AGE LOTID 11144 -0.16769671 0.19020538 AGE LOTID 11144 0.01415533 0.01316629 INTERCEPT LOTID 11145 -0.09516341 0.19031856 AGE LOTID 11145 -0.09516341 0.19031856 AGE LOTID 11145 -0.09516341 0.19031856 AGE LOTID 11146 -0.08786149 0.18872403 AGE LOTID 11146 -0.08786149 0.18872403 AGE LOTID 11147 -0.21248452 0.18231704 AGE LOTID 11147 -0.21248452 0.18231704 AGE LOTID 11148 -0.11397421 0.18758544 AGE LOTID 11148 -0.11397421 0.18758544 AGE LOTID 11148 -0.07768438 0.18631350 AGE LOTID 11149 -0.07768438 0.18631350 AGE LOTID 11149 -0.07768438 0.18631350 AGE LOTID 11150 -0.15424392 0.18762834 AGE LOTID 11150 -0.15424392 0.18762834 AGE LOTID 11151 -0.12749461 0.18679008 AGE LOTID 11151 -0.12749461 0.18679008 AGE LOTID 11156 -0.0861269 0.01295106 INTERCEPT LOTID 11156 -0.0861269 0.01295106 INTERCEPT LOTID 11157 -0.17588616 0.185506946 AGE LOTID 11158 -0.13427054 0.18506946 AGE LOTID 11159 -0.2254272 0.18296667 INTERCEPT LOTID 11158 -0.12427054 0.18506946 AGE LOTID 11159 -0.22564272 0.18296667 INTERCEPT LOTID 11158 -0.12427054 0.18506946 AGE LOTID 11159 -0.22564272 0.18296667 INTERCEPT LOTID 11158 -0.12427054 0.18506946 AGE LOTID 11159 -0.22564272 0.18279375 INTERCEPT LOTID 11181 -0.16269825 0.18241499 AGE LOTID 11180 -0.0759863 0.01279735 INTERCEPT LOTID 11181 -0.16269825 0.18241499 AGE LOTID 11181 -0.16269825 0.18241499 AGE LOTID 11180 -0.01251881 0.012966667 AGE LOTID 11181 -0.126297 0.01279876 INTERCEPT LOTID 11184 0.00759863 0.01279735 INTERCEPT LOTID 11184 0.22032950 0.18650853 INTERCEPT LOTID 11180 0.00759863 0.01279735 INTERCEPT LOTID 11181 0.00759863 0.01279735 INTERCEPT LOTID 11180 0.00759863 0.01279735 INTERCEPT LOTID 11180 0.00759863 0.01279735 INTERCEPT LOTID 11180 0.01252880 0.013305212 INTERCEPT LOTID 11188 0.024783741 0.18692804 AGE LOTID 11188 0.024783741 0.18692804 AGE LOTID 11189 0.22284679 0.18633335 AGE LOTID 11189 0.22284679 0.18	PARAMETER	SUB	JECT	ESTIMATE	STD ERROR
AGE	INTERCEPT	LOTID	11142	-0.06196541	0.19329612
AGE	AGE	LOTID	11142	0.00479259	0.01346718
INTERCEPT	INTERCEPT	LOTID	11143	-0.11611040	0.18778713
INTERCEPT	AGE	LOTID	11143	0.01007674	0.01290832
AGE	INTERCEPT		11144	-0.16769671	0.19020538
AGE LOTID 11145	AGE	LOTID	11144	0.01415533	0.01316629
INTERCEPT	INTERCEPT	LOTID	11145	-0.09516341	0.19031856
AGE LOTID 11146	AGE	LOTID	11145	0.00494655	0.01335297
INTERCEPT LOTID 11147	INTERCEPT	LOTID	11146	-0.08786149	0.18872403
AGE LOTID 11147	AGE	LOTID	11146	0.00599071	0.01323274
INTERCEPT	INTERCEPT	LOTID	11147	-0.21248452	0.18231704
AGE	AGE	LOTID	11147	0.01167817	0.01275956
INTERCEPT LOTID 11149 -0.07768438 0.18631350 AGE LOTID 11149 -0.00123725 0.01287421 INTERCEPT LOTID 11150 -0.15424392 0.18762834 AGE LOTID 11150 0.00950736 0.01304139 INTERCEPT LOTID 11151 -0.12749461 0.18679008 AGE LOTID 11151 0.00861269 0.01295106 INTERCEPT LOTID 11156 -0.15799910 0.18658553 AGE LOTID 11156 0.00818934 0.01299146 INTERCEPT LOTID 11157 -0.17588616 0.18545442 AGE LOTID 11157 -0.17588616 0.18545442 AGE LOTID 11158 -0.13427054 0.18650946 INTERCEPT LOTID 11158 0.00509910 0.01293535 INTERCEPT LOTID 11159 -0.23813672 0.18472938 AGE LOTID 11159 0.01251881 0.01296406 INTERCEPT LOTID 11160 -0.22564272 0.18029667 AGE LOTID 11160 -0.22564272 0.18029667 AGE LOTID 11181 0.00759863 0.01279735 INTERCEPT LOTID 11181 0.00759863 0.01279735 INTERCEPT LOTID 11182 -0.14882165 0.18454494 AGE LOTID 11183 0.17988730 0.18660005 AGE LOTID 11183 0.17988730 0.18660005 AGE LOTID 11184 -0.01074349 0.01305771 INTERCEPT LOTID 11184 -0.01325950 0.18618665 AGE LOTID 11184 -0.01325950 0.18618665 AGE LOTID 11185 0.44493213 0.19631611 AGE LOTID 11188 0.24783741 0.18929810 AGE LOTID 11188 0.24783741 0.18929810 AGE LOTID 11189 -0.001325530 0.01315288 INTERCEPT LOTID 11189 -0.01325530 0.01330528 INTERCEPT LOTID 11189 -0.01325530 0.01330528 INTERCEPT LOTID 11189 -0.01325530 0.01330528 INTERCEPT LOTID 11189 -0.001325530 0.013310098	INTERCEPT	LOTID	11148	-0.11397421	0.18758544
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AGE	LOTID 11192	-0.01170315	0.01302592
INTERCEPT	LOTID 11193	0.19615700	0.18504953
AGE	LOTID 11193	-0.01181337	0.01297891
INTERCEPT	LOTID 11194	0.17762001	0.18452648
AGE	LOTID 11194	-0.01152574	0.01310043
INTERCEPT	LOTID 11195	0.16493381	0.18380045
AGE	LOTID 11195	-0.01078221	0.01291217

Table 9. $\hat{\mathbf{v}}$ And Std Error of $\hat{\mathbf{v}}$

Subsequently, the safe shelf-life of the group of similar lots (t_{sl}) that takes into account the lot-to-lot variation can be obtained as follows:

$$30 = (1, t_{sl}) \begin{pmatrix} 3.81021477 \\ -0.14105394 \end{pmatrix} - t(0.95, N-2) SEPRED_{(group)} \quad (\tilde{y} \mid t_{sl})^{(36)}$$

Figure C.22 shows the resulting shelf-life is 8405.12 days (23.02 years).

This result can be compared to the one obtain by the current method (5), which is 8469 days (23.20 years). Note that the current method without taking into account the potential lot-to-lot variation provides a liberal shelf-life that is 64 days longer than the random effects regression model.

Finally, individual shelf-life of lot i (t_{ssl}) based on the shrinkage estimation (28) is computed by checking the time period at which the expected fume time curve intersects the 30 days of fume time (see Table 10). The resulting individual shelf-life (t_{ssl}) varies from 27.998 to 24.942 years. They all exceed the group shelf-life based on either the fixed effects (23.20 years) or the random effects (23.02 years) regression model.

The group shelf-lives based on the prediction limit for the fume time can be compared to those based on the confidence limit for the expected value. They turn out to be 25.66 and

ОВ	S LOTID	shelf-life (shrinkage (estimation)		
1	11142	26.914	24.520	23.427
2	11143	27.576	25.131	23.971
	11144	28.056	25.411	24.315
4	11145	26.691	24.365	23.219
5	11146	26.951	24.726	23.424
6	11147	27.173	24.729	23.561
7	11148	27.272	24.863	23.698
8	11149	25.654	23.630	22.459
9	11150	27.167	24.726	23.561
10	11151	27.185	24.794	23.629
11	11156	27.074	24.589	23.356
	11157	27.998	25.411	24.246
	11158	26.433	24.178	23.013
	11159	27.151	24.522	23.492
	11160	27.600	25.069	23.904
16	11181	26.715	24.383	23.218
17	11182	27.576	25.068	23.972
18	11183	25.563	23.767	22.663
	11184	25.534	23.629	22.552
20	11185	24.942	23.212	22.191
21	11188	25.526	23.631	22.573
22	11189	25.603	23.698	22.621
23	11190	25.275	23.493	22.459
24	11191	25.555	23.632	22.578
	11192	25.772	23.835	22.735
	11193	25.672	23.771	22.658
27	11194	25.590	23.561	22.550
28	11195	25.639	23.698	22.614

Table 10. Individual Shelf-Life of Propellant Lot

24.314 years, which are longer than their counterparts based on the prediction limit.

The individual shelf-lives can also be calculated by checking the time period when 95% one-sided lower prediction or confidence limits intersect 30 days of fume time. The results are shown in Table 10. About 42% of individual shelf-lives based on the prediction limit turned out to be shorter

than the group shelf-life (23.02 years). The rest are as same as the shrinkage estimation that all exceeds the group shelf-life based on either the fixed and random effects regression model.

All calculations are done by using PROC MIXED and PROC REG of a statistical package SAS [Ref.12].

IV. CONCLUSIONS AND RECOMMENDATIONS

A. CONCLUSIONS

Most gun propellant is stored at depots for a long time before it is used. In storage, the quality of the gun propellant may deteriorate and become unstable. In an attempt to avoid disaster due to use of unstable gun propellant, accurate prediction of the safe shelf-life of gun propellant The shelf-life estimation methods used is necessary. currently for a group of similar gun propellant lots are based on a fixed effects regression model. This does not take into consideration the fact that samples from the same lot are more similar than samples between lots. To capitalize on this lotto-lot variation when estimating the shelf-life, first, a random effects regression model (RERM) is developed. Secondly, a combined mixed effects model is estimated. estimated model is then used to predict not only the shelflife of a group of similar lots but also that of each individual lot of 5"/54 NACO gun propellant stockpile. Finally, results of the data analysis are discussed and recommendations are made.

The claimed shelf-life of the NACO propellant lots provided by NSWC is 35 years. It is interesting to note that when the confidence limit method is used instead of the prediction limit method, the standard linear regression model I provides 35 years as the estimated shelf-life. However, the assumptions for standard linear regression model I were not validated. Therefore, the use of the claimed shelf-life (35 years) is not supported and needs to be amended.

The group shelf-life estimated based on a random effects regression model turns out to be 23.02 years. A fixed effects regression model based on the standard linear regression model II provides a slightly longer shelf-life (23.20 years) than 23.02 years. The individual shelf-lives based on the

shrinkage estimation vary from 27.998 to 24.942 years which all exceed the group shelf-lives obtained from both the fixed and the random effects regression models. It indicates that group shelf-life estimated based on the fixed or the random effects regression models can be relatively conservative compared to the individual shelf-lives. When the group shelf-life (23.02 years) is applied to the maintenance of unstable stockpiles, there is a chance that all individual lots would be discarded even though they are still safe. In view of such opportunity loss, use of individual shelf-life is recommended over the group shelf-life when the management of individual lot is expected to cost less than that of the bulk management.

B. RECOMMENDATIONS

In this thesis, a particular propellant stockpile data set is analyzed. Although the approaches proposed can be applied to any other types of propellant, the model selection may be dictated by the nature of data set.

There is a nonlinear decreasing trend in fume time in some individual lots as the propellant ages. This raises questions about the approaches that use traditional linear least squares analysis of fume time data to predict safe shelf-life. This thesis recommends that the nonlinear regression analysis approach be conducted for some individual propellant lots.

APPENDIX A MANUFACTURING DATE

OBS	LOTID	MANUFACTURING	OBS	LOTID	MANUFACTURING
		DATE			DATE
1	11142	10/16/1972	2	11143	11/06/1972
3	11144	11/07/1972	4	11145	11/28/1972
5	11146	12/07/1972	6	11147	4/01/1973
7	11148	12/27/1972	8	11149	12/27/1972
9	11150	12/27/1972	10	11151	1/24/1973
11	11156	1/24/1973	12	11157	2/15/1973
13	11158	4/01/1973	14	11159	4/01/1973
15	11160	10/18/1973	16	11181	10/19/1973
17	11182	4/01/1973	18	11183	10/24/1973
19	11184	10/25/1973	20	11185	7/26/1973
21	11188	7/29/1973	22	11189	10/31/1973
23	11190	10/31/1973	24	11191	11/01/1973
25	11192	11/06/1973	26	11193	1/14/1974
27	11194	1/14/1974	28	11195	1/14/1974

APPENDIX B COMPUTER PROGRAM LIST

The following computer program is a SAS code.

OPTIONS LS=80;

DATA ALL1;		/*	MAS	TER SAMPI	ES	*/	
INPUT ID= N ;	OB	ID	INDEX	EAGE	EDTF	EYAGE	EYDTF;
CARDS;							
·	1	1	11142	204	756	0.5589	2.07123
	2	1	11142	960	875	2.6301	2.39726
	3	1	11142	1834	1117	5.0247	3.06027
	4	ī	11142	2951	1002	8.0849	2.74521
	5	ī	11142	3952	741	10.8274	2.03014
	6	ī	11142	4692	688	12.8548	1.88493
	7	ī	11142	5380	710	14.7397	1.94521
•	8	1	11142	6238	578	17.0904	1.58356
	9	2	11143	184	724	0.5041	1.98356
	10	2	11143	908	837	2.4877	2.29315
	11	2	11143	1744	1083	4.7781	2.96712
	12	2	11143	2827	1058	7.7452	2.89863
	13	2	11143	3884	761	10.6411	2.08493
	14	2	11143	4644	699	12.7233	1.91507
	15	2	11143	5343	727	14.6384	1.99178
	16	2	11143	6218	589	17.0354	1.61370
	17	2	11143	6807	552	18.6493	1.51233
	18	3	11144	276	663	0.7562	1.81644
	19	3	11144	939	833	2.5726	2.28219
	20	3	11144	1771	1077	4.8521	2.95068
	21	3	11144	2848	1043	7.8027	2.85753
	22	3	11144	3890	742	10.6575	2.03288
	23	3	11144	4631	683	12.6877	1.87123
	24	3	11144	5314	685	14.5589	1.87671
	25	3	11144	5998	552	16.4329	1.51233
	26	3	11144	6363	806	17.4329	2.20822
	27	4	11144	913	808	2.5014	2.21370
	28	4	11145	1720	1077	4.7123	2.95068
	29	4	11145	2797	1046	7.6630	2.86575
	30	4	11145	3842	764	10.5260	2.00375
	31	4	11145	4605	683	12.6164	1.87123
	32	4	11145	5288	613	14.4877	1.67945
	33	4	11145	5900	559	16.1644	1.53151
	34	5	11146	152	689	0.4164	1.88767
	35	5	11146	841	834	2.3041	2.28493
	36	5	11146	1674	1045	4.5863	2.86301
	37	5	11146	2719	1106	7.4493	3.03014
	38	5	11146	3824	777	10.4767	2.12877
	39	5	11146	4600	694	12.6027	1.90137
	40	5	11146	5294	717	14.5041	1.96438
	41	5	11146	6010	556	16.4658	1.52329
	42	6	11147	0	661	0.0000	
	43	6	11147	661	855	1.8110	1.81096 2.34247
	44	6	11147	1515	1017	4.1507	2.78630
	45	6	11147	2532	1017	6.9370	2.89589
	46	6	11147	3588	786	9.8301	2.15342
	47	6	11147	4373	700	11.9808	1.91781
	- /	3	-	43/3	700	11.9000	1.71/01

4.0	_	11147	5073	655	13.8986	1.79452
48	6				15.6904	1.53425
49	6	11147	5727	560		
50	6	11147	6287	459	17.2247	1.25753
51	7	11148	132	728	0.3616	1.99452
5 2	7	11148	857	834	2.3479	2.28493
5 3	7	11148	1690	1077	4.6301	2.95068
54	7	11148	2767	1065	7.5808	2.91781
	7	11148	3831	761	10.4959	2.08493
55					12.5781	1.93973
56	7	11148	4591	708		
57	7	11148	5299	696	14.5178	1.90685
58	7	11148	5994	528	16.4219	1.44658
59	7	11148	6522	596	17.8685	1.63288
60	8	11149	888	806	2.4329	2.20822
61	8	11149	1693	1046	4.6384	2.86575
62	8	11149	2739	1086	7.5041	2.97534
63	8	11149	3824	737	10.4767	2.01918
				699	12.4932	1.91507
64	8	11149	4560			
65	8	11149	5259	637	14.4082	1.74521
66	8	11149	5895	19	16.1507	0.05205
67	8	11149	6813	477	18.6658	1.30685
68	9	11150	132	728	0.3616	1.99452
69	9	11150	860	824	2.3562	2.25753
70	9	11150	1683	1063	4.6110	2.91233
			2746	1074	7.5233	2.94247
71	9	11150				
72	9	11150	3819	728	10.4630	1.99452
73	9	11150	4546	682	12.4548	1.86849
74	9	11150	5228	512	14.3233	1.40274
75	9	11150	5740	702	15.7260	1.92329
76	9	11150	6441	538	17.6466	1.47397
7 7	10	11151	844	823	2.3123	2.25479
78	10	11151	1666	1056	4.5644	2.89315
79	10	11151	2722	1083	7.4575	2.96712
	10	11151	3804	747	10.4219	2.04658
80					12.4658	1.91233
81	10	11151	4550	698		
82	10	11151	5248	711	14.3781	1.94795
83	10	11151	5958	52 3	16.3233	1.43288
84	10	11151	6481	557	17.7562	1.52603
85	11	11156	106	709	0.2904	1.94247
86	11	11156	815	842	2.2329	2.30685
87	11	11156	1656	1038	4.5370	2.84384
88	11	11156	2694	1062	7.3808	2.90959
			3755	765	10.2877	2.09589
89	11	11156		665	12.3808	1.82192
90	11	11156	4519			
91	11	11156	5184	589	14.2027	1.61370
92	11	11156	5773	592	15.8164	1.62192
93	11	11156	6364	463	17.4356	1.26849
94	12	11157	822	822	2.2521	2.25205
95	12	11157	1643	1084	4.5014	2.96986
96	12	11157	2727	1056	7.4712	2.89315
97	12	11157	3782	746	10.3616	2.04384
98	12	11157	4527	710	12.4027	1.94521
	12	11157	5237	723	14.3479	1.98082
99			6118	563	16.7616	1.54247
100	12	11157				
101	12	11157	6681	662	18.3041	1.81370
102	13	11158	0	738	0.0000	2.02192
103	13	11158	738	829	2.0219	2.27123
104	13	11158	1566	1092	4.2904	2.99178
105	13	11158	2658	1041	7.2822	2.85205
106	13	11158	3698	746	10.1315	2.04384
107	13	11158	4443	681	12.1726	1.86575
108	13	11158	5124	620	14.0384	1.69863

109	13	11158	5743	448	15.7342	1.22740
110	13	11158	6191	482	16.9616	1.32055
111	14	11159	0	730	0.0000	2.00000
112	14	11159	730	842	2.0000	2.30685
113	14	11159	1571	1116	4.3041	3.05753
114	14	11159	2687	1042	7.3616	2.85479
115	14	11159	3728	550	10.2137	1.50685
116	14	11159	4277	624	11.7178	1.70959
117	14	11159	4901	661	13.4274	1.81096
118	14	11159	5562	666	15.2384	1.82466
119	14	11159	6227	489	17.0603	1.33973
120	15	11160	582	834	1.5945	2.28493
121	15	11160	1415	1085	3.8767	2.97260
122	15	11160	2500	1038	6.8493	2.84384
123	15	11160	3537	745	9.6904	2.04110
124	15	11160	4281	695	11.7288	1.90411
125	15	11160	4976	720	13.6329	1.97260
126	15	11160	5695	573	15.6027	1.56986
127	15	11160	6268	581	17.1726	1.59178
128	16	11181	632	826	1.7315	2.26301
129	16	11181	1457	1177	3.9918	3.22466
130	16	11181	2633	953	7.2137	2.61096
131	16	11181	3586	721	9.8247	1.97534
132	16	11181	4306	680	11.7973	1.86301
133	16	11181	4986	686	13.6603	1.87945
134	16	11181	5671	524	15.5370	1.43562
135	16	11181	6195	488	16.9726	1.33699
136	17	11182	0	741	0.0000	2.03014
137	17	11182	741	841	2.0301	2.30411
138	17	11182	1581	1131	4.3315	3.09863
139	17	11182	2712	1023	7.4301	2.80274
140	17	11182	3734	747	10.2301	2.04658
141	17	11182	4480	691	12.2740	1.89315
142	17	11182	5171	716	14.1671	1.96164
143	17	11182	6008	595	16.4603	1.63014
144	17	11182	6603	600	18.0904	1.64384
145	18	11183	622	840	1.7041	2.30137
146	18	11183	1461	1300	4.0027	3.56164
147	18	11183	2760	1105	7.5616	3.02740
148	18	11183	3865	779	10.5890	2.13425
149	18	11183	4643	734	12.7205	2.01096
150	18	11183	5377	713	14.7315	1.95342
151	18	11183	6089	539	16.6822	1.47671
152	19	11184	586	846	1.6055	2.31781
153	19	11184	1431	1322	3.9205	3.62192
154	19	11184	2752	1112	7.5397	3.04658
155	19	11184	3864	781	10.5863	2.13973
156	19	11184	4644	736	12.7233	2.01644
157	19	11184	5380	692	14.7397	1.89589
158	19	11184	6071	515	16.6329	1.41096
159	20	11185	0	698	0.0000	1.91233
160	20					
		11185	698	1049	1.9123	2.87397
161	20	11185	1746	1492	4.7836	4.08767
162	20	11185	3237	998	8.8685	2.73425
163	20	11185	4234	784	11.6000	2.14795
164	20	11185	5018	766	13.7479	2.09863
165	20					
		11185	5783	569	15.8438	1.55890
166	20	11185	6352	579	17.4027	1.58630
167	21	11188	0	684	0.0000	1.87397
168	21	11188	684	854	1.8740	2.33973
169	21	11188	1537	1358	4.2110	3.72055
						5.72055

	170	21	111	188	2894	1072	7.9288	2.93699	
	171	21		188	3966	766	10.8658	2.09863	
	172	21		188	4731	743	12.9616	2.03562	
	173	21		188	5474	689	14.9973	1.88767	
	174	21		188	6162	553	16.8822	1.51507	
	175	22		189	597	831	1.6356	2.27671	
	176	22		189	1427	1351	3.9096	3.70137	
	177	22		189	2777	1085	7.6082	2.97260	
	178	22		189	3862	795	10.5808	2.17808	
		22		189	4656	740	12.7562	2.02740	
	179 180	22		189	5396	695	14.7836	1.90411	
				189	6090	564	16.6849	1.54521	
	181 182	22 23		190	596	866	1.6329	2.37260	
	183	23		190	1461	1316	4.0027	3.60548	
		23		190	2776	1075	7.6055	2.94521	
	184	23		190	3851	798	10.5507	2.18630	
	185 186	23		190	4648	719	12.7342	1.96986	
				190	5367	586	14.7041	1.60548	
	187	23		190	5952	540	16.3068	1.47945	
	188	23 24		191	601	825	1.6466	2.26027	
	189			191	1425	1349	3.9041	3.69589	
	190	24			2773	1096	7.5973	3.00274	
	191	24		191	3869	770	10.6000	2.10959	
	192	24		191	4638	770 757	12.7068	2.07397	
	193	24		191		683	14.7808	1.87123	
	194	24		191	5395 6077	553	16.6493	1.51507	
	195	24		191	598	824	1.6384	2.25753	
	196	25		192			3.8932	3.71781	
	197	25		192	1421	1357 1069	7.6082	2.92877	
	198	25		192	2777		10.5370	2.18904	
	199	25		192	3846	799 746	12.7233	2.16904	
	200	25		192	4644		14.7671	1.91781	
	201	25		192	5390	700	16.6822	1.68493	
	202	25		192	6089	615 051			
	203	26		193	528	851	1.4466 3.7753	2.33151 3.72877	
	204	26		193	1378	1361	7.5014	2.94795	
	205	26		193	2738	1076		2.09315	
	206	26		193	3814	764	10.4493 12.5397	2.05205	
	207	26		193	4577 5326	749 699	14.5918	1.91507	
	208	26		193				1.60274	
	209	26		193	6024	585	16.5041	2.26027	
	210	27		194	528	825	1.4466	3.64110	
	211	27		194	1352 2680	1329 1075	3.7041 7.3425	2.94521	
	212	27		194			10.2877	2.26849	
	213	27		194	3755	828		2.28849	
	214	27		194	4582	742	12.5534	2.25479	
	215	28		195	528	823 1331	3.6986	3.64658	
	216	28		195	1350	1059	7.3425	2.90137	
	217	28		195	2680			2.21370	
	218	28		195	3739	808	10.2438	2.06301	
	219	28		195	4546	753 604	12.4548		
	220	28		195	5299	694	14.5178	1.90137	
	221	28	11.	195	5992	507	16.4164	1.38904	
					/* FI	EET RETURN	DATA	*/	
DATA ALL;					,			,	
INPUT			OBS	ID	INDEX	MDATE	DTF	AGE	;
CARD;			-	-	11110	70 0705	77.6	1671 0	
			1	1	11142	72.8795	716	1671.0	
			2 3	1 1	11142 11142	72.8795 72.8795	662 812	4372.0 1671.0	
			ے	Τ.	11142	12.0133	012	10/1.0	

4	1	11142	72.8795	923	1802.0
5	1	11142	72.8795	965	1802.0
6	1	11142	72.8795	665	361.0
7	1	11142	72.8795	769	2364.0
8	1	11142	72.8795	738	1401.0
9	1 1	11142	72.8795	716 479	1671.0 1183.0
10 11	1	11142 11142	72.8795 72.8795	734	2426.0
12	1	11142	72.8795	785	2749.0
13	1	11142	72.8795	685	2896.0
14	1	11142	72.8795	716	1671.0
15	1	11142	72.8795	718	4372.0
16	1	11142	72.8795	795	1802.0
17 18	2 3	11143 11144	72.8521 72.9384	722 737	1812.0 779.5
19	3	11144	72.9384	662	1427.5
20	3	11144	72.9384	814	1521.5
21	3	11144	72.9384	639	5054.5
22	3	11144	72.9384	638	4350.5
23	4	11145	72.9959	719	2325.5
24 25	4 4	11145 11145	72.9959 72.9959	611 723	5074.5 1358.5
26	4	11145	72.9959	812	1628.5
27	4	11145	72.9959	681	2462.5
28	4	11145	72.9959	648	4329.5
29	4	11145	72.9959	697	2788.5
30	4	11145	72.9959	698	2788.5
31	4	11145	72.9959	682 693	2810.5 2810.5
32 33	4 4	11145 11145	72.9 9 59 72. 995 9	798	1628.5
34	4	11145	72.9959	623	4329.5
35	4	11145	72.9959	795	1759.5
36	4	11145	72.9959	686	1759.5
37	5	11146	73.0219	614	4360.0
38	5 5	11146 11146	73.0219 73.0219	628 611	4320.0 4337.0
39 40	5 5	11146	73.0219	618	5024.0
41	5	11146	73.0219	643	2123.0
42	5	11146	73.0219	781	1619.0
43	6	11147	73.3370	847	1016.0
44	6	11147	73.3370	710	2259.0
45 46	6 6	11147 11147	73.3370 73.3370	635 614	4205.0 4205.0
47	6	11147	73.3370	722	1635.0
48	6	11147	73.3370	667	2706.0
49	6	11147	73.3370	673	2706.0
50	6	11147	73.3370	613	4950.0
51 52	6 6	11147 11147	73.3370 73.3370	525 623	2139.0 2008.0
53	6	11147	73.3370	677	2197.0
54	7	11148	73.0767	611	5004.0
55	7	11148	73.0767	703	2824.0
56	7	11148	73.0767	728	2354.0
57	7	11148	73.0767	626	5004.0
58 59	7 7	11148 11148	73.0767 73.0767	736 1965	1730.0 1730.0
60	7	11148	73.0767	575	2234.0
61	7	11148	73.0767	667	2801.0
62	7	11148	73.0767	673	2801.0
63	7	11148	73.0767	683	2801.0
64	7	11148	73.0767	690	2801.0

65 66 67 71 72 73 74 75 77 78 79 81 82 83 84 85 87 77 78 79 80 81 82 83 84 85 86 87 88 89 99 10 99 99 99 99 10 10 10 10 10 10 10 10 10 10	7 11148 7 11148 7 11148 7 11148 8 11149 8 11149 8 11149 11150 11150 11150 11150 11150 11151 11151 11151 11151 11151 11151 11151 11151 11151 11151 11151 11151 11151 11157 11157 11157 11157 11157 11157 11157 11157 11157 11157 11158 11158 11158 11158 11158 11158 11158 11158 11159 11159 11160 11160	73.0767 73.0767 73.0767 73.0767 73.0767 73.0767 73.0767 73.0767 73.0767 73.0767 73.0767 73.0767 73.0767 73.0767 73.0767 73.1493	7991531881673664849521886076502840475303287726473776111348827726473775985260487776111348827726447337759852604822	2801.0 1593.0 4300.0 28299.0 4300.0 5045.0 2801.0 2801.0 2801.0 2801.0 2801.0 2801.0 2801.0 2801.0 2801.0 2801.0 2801.0 2801.0 2103.0 4300.0 4300.0 4300.0 2103.0 4300.
	11160 11160		672 677 690	1435.0 1082.0 4005.0 4005.0

126 127 128 129 130 131 132 133 134 135	15 16 16 17 17 17 17 17	11160 11181 11181 11182 11182 11182 11182 11182 11182 11182	73.8849 73.8877 73.8877 73.8877 73.3370 73.3370 73.3370 73.3370 73.3370 73.3370 73.3370	930 634 672 742 624 624 627 930 707 711	724.0 4708.0 1081.0 1434.0 4909.0 4909.0 4222.0 924.0 2664.0
137 138 139 140 141 142 143 144 145	17 17 18 18 18 18 18 19	11182 11183 11183 11183 11183 11183 11183 11184 11184	73.3370 73.3370 73.9014 73.9014 73.9014 73.9014 73.9014 73.9014 73.9041 73.9041	715 706 808 682 693 668 623 993 838 943	2664.0 2686.0 428.0 3999.0 1076.0 3999.0 4744.0 718.0 427.0 717.0
147 148 149 150 151 152 153 154	20 20 20 21 21 21 21 21	11185 11185 11185 11185 11188 11188 11188 11188	73.6562 73.6562 73.6562 73.6562 73.6644 73.6644 73.6644 73.6644 73.6644	818 657 703 719 662 694 716 662 781	517.5 4792.5 2214.5 1165.5 4085.5 1162.5 1384.5 4085.5 2019.5
156 157 158 159 160 161 162 163 164 165	22 22 22 22 22 22 22 22 22 22	11189 11189 11189 11189 11189 11189 11189 11189	73.9178 73.9178 73.9178 73.9178 73.9178 73.9178 73.9178 73.9178 73.9178 73.9178	624 706 662 618 819 829 685 792 716	4697.0 2126.0 3993.0 4697.0 1292.0 1160.0 1927.0 1989.0 1292.0 3993.0
166 167 168 169 170 171 172 173 174	23 23 23 23 23 24 24 24 24	11190 11190 11190 11190 11190 11191 11191 11191	73.9205 73.9205 73.9205 73.9205 73.9205 73.9219 73.9219 73.9219 73.9219	818 791 709 690 869 1336 716 648 877	1422.0 1647.0 2493.0 3992.0 1422.0 1794.5 1290.5 3991.5 1421.5
175 176 177 178 179 180 181 182 183 184 185 186	24 24 24 24 24 24 25 25 25 26	11191 11191 11191 11191 11191 11191 11192 11192 11192 11192 11193	73.9219 73.9219 73.9219 73.9219 73.9219 73.9219 73.9219 73.9356 73.9356 73.9356 73.9356 74.1219	925 708 709 707 594 690 653 638 683 700 706	1421.5 2450.5 2450.5 2472.5 4695.5 2124.5 3991.5 4690.5 1063.5 3873.5 3805.5

```
74.1219
                                                 702
                                                        2399.5
                     187 26 11193
                        26
                             11193
                                                 703
                     188
                                      74.1219
                                                        2399.5
                             11193
                                      74.1219
                                                 710
                                                        2399.5
                     189
                         26
                     189 26
190 26
                                      74.1219
                                                 726
                             11193
                                                        2399.5
                     191 26
                             11193
                                      74.1219
                                                 918
                                                        637.5
                     192 26 11193
                                      74.1219
                                                 711
                                      74.1219
                                                 725
                     193 26 11193
                     194 26 11193
                                      74.1219
                                                726
                                      74.1219
                                                 731
                     195 26 11193
                                                       2399.5
                                      74.1219 697
                     196 26 11193
                                                        2044.5
                                      74.1219
                         26 11193
                                                 700
                     197
                                                        2051.5
                         26 11193
                                      74.1219
                                                        1217.5
                     198
                                                 716
                         26
                                      74.1219
                                                 654
                     199
                             11193
                                                        3918.5
                                      74.1219
                         26
                             11193
                                                 646
                                                        3918.5
                     200
                                      74.1219
                         27 11194
                                                 647
                                                       4622.5
                     201
                         27 11194
                                      74.1219
                                                688
                                                     2044.5
                     202
                     203 28 11195
                                     74.1219 612
                                                      4622.5
                     204 28 11195
                                     74.1219 609 4622.5
PROC SORT: BY INDEX;
PROC PRINT DATA= ALL1; VAR INDEX EAGE EDTF EYAGE EYDTF;
               COMBINE MASTER SAMPLE AND FLEET RETURN DATA
DATA NEW;
MERGE ALL1 ALL2 ; BY INDEX;
IF ID = LAG(ID) THEN EAGE = .;
IF ID = LAG(ID) THEN EDTF = .;
PROC PRINT DATA=NEW; VAR INDEX MDATE DTF AGE EDTF EAGE;
PROC UNIVARIATE;
PROC PLOT;
     PLOT DTF*AGE='A' EDTF*EAGE='*';
PROC PLOT;
     PLOT DTF*AGE='A' EDTF*EAGE='*'/OVERLAY;
                                                         */
          /*
               STANDARD LINEAR REGRESSION MODEL I
PROC REG ; MODEL DTF = AGE ;
               STANDARD LINEAR REGRESSION MODEL II
         /*
                   ( DELET EAGE BELOW 1100 DAYS )
DATA ALL3 (DROP= OB ID EYAGE EYDTF); SET ALL1;
DATA ALL4; SET ALL3;
IF EAGE LE 1100 THEN DELETE;
PROC PLOT ;
     PLOT EDTF*EAGE='*';
PROC REG ; MODEL EDTF= EAGE;
OUTPUT OUT=OUT1 P= PRED R=RES;
PROC PRINT;
DATA ALL5 (DROP= RES ) ; MERGE ALL4 OUT1; BY INDEX;
PROC PRINT;
DATA ALL6(DROP=OBS); SET ALL5; BY INDEX;
W = (EAGE - 4146.15) * (EAGE - 4146.15);
SXX = 2521553*(180-1);
SEPRED = 98.295 * SQRT(1+(1/180)+(W/SXX));
L95=PRED-1.645*SEPRED;
```

```
U95=PRED+1.645*SEPRED;
EAGE30=30;
PROC PRINT;
PROC PLOT ;
     PLOT EDTF*EAGE='A' PRED*EAGE='-' L95*EAGE='.'
          U95*EAGE='.' EAGE30*EAGE='-'/OVERLAY;
     PLOT PRED*EAGE='*' L95*EAGE='.'
          U95*EAGE='.' EAGE30*EAGE='-'/OVERLAY;
DATA EXTEN1;
DO DAGE = 8000 TO 12000 BY 100;
PRED=1045.510-0.07*EAGE;
W=(EAGE-4146.15)*(EAGE-4146.15);
SXX= 2521553*(180-1);
SEPRED = 98.295 * SQRT(1+(1/180)+(W/SXX));
L95=PRED-1.645*SEPRED;
U95=PRED+1.645*SEPRED;
OUTPUT;
END;
PROC PRINT DATA=EXTEN1; VAR EAGE PRED L95 U95;
                    LINEARIZING TRANSFORMATION MODEL I
                           (TRANSFER EAGE TO 1/EAGE)
DATA ALL7 ; SET ALL1;
IF EAGE LE 1100 THEN DELETE;
Y=1/EAGE;
PROC REG ; MODEL EDTF= Y;
OUTPUT OUT=OUT2 P= PRED R=RES STDI=SEP;
PROC PLOT;
     PLOT EDTF*EAGE;
DATA ALL8 (DROP= RES ) ; MERGE ALL7 OUT2; BY INDEX;
PROC UNIVARIATE;
DATA ALL9 (DROP=OBS); SET ALL8; BY INDEX;
W = (Y-0.000297) * (Y-0.000297);
SXX= 0.000164*0.000164*(180-1);
SEPRED = 112.854* SQRT(1+(1/180)+(W/SXX));
L95=PRED-1.645*SEPRED;
U95=PRED+1.645*SEPRED;
PROC PRINT DATA=ALL9 ; VAR INDEX EAGE EDTF PRED U95 L95;
PROC PLOT :
 PLOT EDTF*Y='A' PRED*EAGE='-' L95*EAGE='.'
          U95*EAGE='.' /OVERLAY;
     PLOT PRED*EAGE='*' L95*EAGE='.'
          U95*EAGE='.' /OVERLAY;
 DATA EXTEN2;
 DO EAGE=15000 TO 35000 BY 100;
 PRED=411.737+1315827*(1/EAGE);
W=((1/EAGE)-0.000297)*((1/EAGE)-0.000297);
SXX= 0.000164*0.000164*(180-1);
SEPRED = 112.854* SQRT(1+(1/180)+(W/SXX));
L95=PRED-1.645*SEPRED;
U95=PRED+1.645*SEPRED;
```

```
OUTPUT;
END;
PROC PRINT DATA=EXTEN2; VAR EAGE PRED L95 U95;
                 LINEARIZING TRANSFORMATION MODEL II
                      (TRANSFER EDTF TO LOG(EDTF))
DATA ALL10; SET ALL1;
IF EAGE LE 1100 THEN DELETE;
EDTFLOG = LOG(EDTF);
PROC REG ; MODEL EDTFLOG = EAGE;
OUTPUT OUT=OUT3 P= PRED R=RES STDI=SEP;
PROC PLOT;
     PLOT EDTF*EAGE;
DATA ALL11 (DROP= RES ) ; MERGE ALL10 OUT3; BY INDEX;
PROC UNIVARIATE;
DATA ALL12 (DROP=OBS); SET ALL11; BY INDEX;
W = (EAGE - 4146) * (EAGE - 4146);
SXX= 2521553*(180-1);
SEPRED = 0.2742 * SQRT(1+(1/180)+(W/SXX));
L95=PRED-1.645*SEPRED;
U95=PRED+1.645*SEPRED;
PROC PRINT DATA=ALL12 ; VAR INDEX EAGE EDTF PRED L95 U95;
PROC PLOT ;
     PLOT EDTFLOG*EAGE='A' PRED*EAGE='-' L95*EAGE='.'
          U95*EAGE='.' /OVERLAY;
     PLOT PRED*EAGE='*' L95*EAGE='.'
          U95*EAGE='.' /OVERLAY;
 DATA EXTEN3;
 DO EAGE=14000 TO 20000 BY 100;
 EDTFH=EXP(7.3787-0.000197*EAGE);
 PRED =7.3787-0.000197*EAGE;
W = (EAGE - 4146) * (EAGE - 4146);
SXX = 2521553*(180-1);
SEPRED = 0.2742 * SORT(1+(1/180)+(W/SXX));
L95=PRED-1.645*SEPRED;
U95=PRED+1.645*SEPRED;
EXPL95=EXP(PRED-1.645*SEPRED);
EXPU95=EXP(PRED+1.645*SEPRED);
OUTPUT:
END;
PROC PRINT DATA=EXTEN3; VAR EAGE PRED L95 U95 EXPL95;
          /*
                    FLEET RETURN DATA
                                                 */
DATA ALL14 ;SET ALL2;
IF DTF GE 1200 THEN DELETE;
PROC PLOT;
    PLOT DTF*AGE='*';
PROC REG ; MODEL DTF= AGE;
OUTPUT OUT=OUT5 P= PRED R=RES STDI=SEP;
```

```
PROC PRINT;
PROC UNIVARIATE;
DATA ALL15 (DROP= RES ) ; MERGE ALL14 OUT5; BY INDEX;
PROC PRINT DATA = ALL15; VAR INDEX DTF AGE PRED;
PROC PRINT ;
DATA ALL16; SET ALL15; BY INDEX;
W = (AGE - 2821.968) * (AGE - 2821.968);
SXX= 1928683*(202-1);
SEPRED = 61.32 * SQRT(1+(1/202)+(W/SXX));
L95=PRED-1.645*SEPRED;
U95=PRED+1.645*SEPRED;
AGE30=30;
PROC PRINT; VAR INDEX AGE DTF PRED U95 L95;
PROC PLOT ;
   PLOT DTF*AGE='A' PRED*AGE='-' L95*AGE='.'
          U95*AGE='.' AGE30*AGE='-'/OVERLAY;
     PLOT PRED*AGE='*' L95*AGE='.'
          U95*AGE='.' AGE30*AGE='-'/OVERLAY;
DATA EXTEN5;
DO AGE=10000 TO 16400 BY 100;
PRED= 823.375-0.0429 *AGE;
W=(AGE-2821.968)*(AGE-2821.968);
SXX= 1928683*(202-1);
SEPRED = 61.32 * SQRT(1+(1/202)+(W/SXX));
L95=PRED-1.645*SEPRED;
TEST=PRED-1.699*SEPRED;
U95=PRED+1.645*SEPRED;
AGE30=30;
OUTPUT;
END;
PROC PRINT DATA= EXTEN5; VAR AGE PRED L95 U95 TEST;
PROC PLOT;
     PLOT L95*AGE='*' AGE30*AGE='.'/OVERLAY;
            /*
                      WEIGHTED LEAST SQUARE MODEL
            /*
                          (DTF/AGE VS 1/AGE)
DATA ALL20 ; SET ALL2;
IF DTF GE 1200 THEN DELETE;
NEWDTF=DTF/AGE ;
NEWAGE=1/AGE;
PROC REG ; MODEL NEWDTF= NEWAGE;
OUTPUT OUT=OUT21 P= PRED R=RES STDI=SEP ;
PROC PRINT;
PROC UNIVARIATE;
DATA ALL22; MERGE ALL20 OUT21; BY INDEX;
       U95 = PRED+1.645*SEP;
       L95 = PRED-1.645*SEP;
PROC PRINT DATA=ALL22; VAR AGE DTF PRED L95 U95 SEP;
PROC PLOT;
```

```
PLOT RES* PRED='*';
  PROC PLOT;
       PLOT L95 *AGE='*' PRED*AGE='*' U95*AGE='*'/OVERLAY;
                      POWER TRANSFORMATION MODEL
              /*
                             (DTF**-3 VS AGE)
  DATA ALL23 ;SET ALL2;
  IF DTF GE 1200 THEN DELETE;
  NEWDTF=DTF**(-3);
  PROC REG ; MODEL NEWDTF= AGE;
  OUTPUT OUT=OUT24 P= PRED R=RES STDI=SEP;
  PROC PRINT;
  PROC UNIVARIATE;
  DATA ALL25; MERGE ALL23 OUT24; BY INDEX;
     U95 = PRED+1.645*SEP;
     L95 = PRED-1.645*SEP;
  PROC PRINT DATA=ALL25; VAR AGE DTF PRED L95 U95 SEP;
  PROC PLOT;
       PLOT RES* PRED='*';
  PROC PLOT;
       PLOT L95 *AGE='*' PRED*AGE='*' U95*AGE='*'/OVERLAY;
  DATA F1M4EXT;
  DO AGE=900000
                 TO 1000000 BY 100;
  PRED= (1.75*(10**(-9)))+(4.87*(10**(-13))) *AGE;
  W=(AGE-2821.968)*(AGE-2821.968);
  SXX= 1928683*(202-1);
  SEPRED = SQRT(6.204807*(10**(-19))) * SQRT(1+(1/202)+(W/SXX));
  U95 = (PRED-1.645*SEPRED)**(-1/3);
  L95 = (PRED + 1.645 * SEPRED) * * (-1/3);
  AGE30=30;
  OUTPUT;
  END;
  PROC PRINT DATA= F1M4EXT; VAR AGE PRED L95 U95 ;
  PROC PLOT;
  PLOT L95*AGE='*' PRED*AGE='.' U95*AGE='[' AGE30*AGE='.'/OVERLAY;
  DATA ONE ( DROP=OBS); SET ALL1;
  FUME=EAGE/365; AGE=EAGE/365;
  PROC PRINT; VAR LOTID AGE FUME;
DATA ONE; SET ALL1; IF AGE < 3 THEN DELETE;
                        /* WITHIN-LOT MODEL */
  PROC MIXED;
  CLASS LOTID;
  MODEL FUME = LOTID AGE*LOTID / NOINT S PREDICTED;
                     /* FIXED EFFECTS REGRESSION MODEL */
  PROC MIXED;
  CLASS LOTID;
  MODEL FUME = AGE / S PREDICTED;
```

```
PROC MIXED ABSOLUTE;
CLASS LOTID;
MODEL FUME = AGE / S PREDICTED;
            AGE / CL G S SUB=LOTID TYPE=UN ;
RANDOM INT
PROC PRINT;
   /* COMPUTE PRED SEPRED U95 L95 FOR RANDOM EFFECTS MODEL
DATA TWO (DROP= OBS); SET ONE; BY LOTID;
PRED=3.81021477-0.14105394 * AGE;
SEPRED=SQRT(( 0.070791)+(2*AGE*(-0.00450653))
           +(AGE*AGE*0.00030661)+(0.05926819));
U95=PRED+1.645*SEPRED;
L95=PRED-1.645*SEPRED;
PREDD=PRED*365;
U95D=U95*365;
L95D=L95*365;
PROC PRINT; VAR LOTID AGE FUME PRED PREDD U95D L95D;
         /* GROUP SHELF-LIFE ESTIMATION */
DATA EXTEN7;
  DO AGE=(5000/365) TO (10000/356) BY (100/365);
     PRED=3.81021477-0.14105394 * AGE;
      SEPRED=SQRT(( 0.070791)+(2*AGE*(-0.00450653))
                  +(AGE*AGE*0.00030661));
     U95=PRED+1.645*SEPRED;
     L95=PRED-1.645*SEPRED;
     PREDD=PRED*365;
     U95D=U95*365;
     L95D=L95*365;
     AGED=AGE*365;
  OUTPUT;
 END;
 PROC PRINT; VAR AGE AGED PRED PREDD L95D
                    /* SHRINKAGE ESTIMATION
                                               */
DATA THREE; INPUT LOTID INTERCEP SLOPE;
CARDS;
                      0.00479259
11142
        -0.06196541
                     0.01007674
11143
       -0.11611040
                      0.01415533
11144
       -0.16769671
11145
       -0.09516341
                     0.00494655
       -0.08786149
                      0.00599071
11146
11147
       -0.21248452
                    0.01167817
       -0.11397421
                    0.00853627
11148
11149
       -0.07768438
                    -0.00123725
       -0.15424392
                     0.00950736
11150
11151
       -0.12749461
                     0.00861269
11156
       -0.15799910
                     0.00818934
```

/* RANDOM EFFECTS REGRESSION MODEL */

0.01418657

11157

-0.17588616

```
-0.13427054
11158
                      0.00509910
        -0.23813672
                      0.01251881
11159
                      0.01415942
11160
        -0.22564272
                     0.00759863
11181
        -0.16269825
                     0.01126227
11182
        -0.14882165
        0.17988730 -0.01074349
11183
        0.21035950 -0.01320753
11184
        0.44493213 -0.02624763
11185
        0.24783741 -0.01470002
11188
        0.22284679 -0.01325530
11189
        0.18150475 -0.01253802
11190
        0.22307183 -0.01355682
11191
                    -0.01170315
        0.20898367
11192
                    -0.01181337
        0.19615700
11193
11194
        0.17762001
                    -0.01152574
        0.16493381 -0.01078221
11195
                    0.00000000
        0.00000000
20000
PROC SORT; BY LOTID;
PROC PRINT;
DATA FOUR; SET THREE ;
INTERC1=INTERCEP + 3.81021477;
SLOPE1=SLOPE-0.14105394;
PROC PRINT; VAR LOTID INTERC1 SLOPE1;
DATA FIVE; SET THREE ;
INTERC1=INTERCEP + 3.81021477;
SLOPE1=SLOPE-0.14105394;
  DO AGE=(5000/365) TO (15000/356) BY (100/365);
      TSSL = INTERC1 + SLOPE1 * AGE ; BY LOTID;
      TSSLD=TSSL*365;
     AGED= AGE*365;
  OUTPUT;
 PROC PRINT; VAR LOTID AGE AGED TSSL TSSLD ;
```

APPENDIX C FIGURES

The figures shown in this appendix are described in Chapter II and IV are as follows :

Figure C.1 -Figure C.3 Standard Linear Regression Model I.

Figure C.4 -Figure C.6 Standard Linear Regression Model II.

Figure C.7 -Figure C.8 Linearizing Transformation Model I.

Figure C.9 -Figure C.11 Linearizing Transformation Model II.

Figure C.12-Figure C.14 Standard Regression Model Based on Fleet return data.

Figure C.15 Weighted Least Square Model.

Figure C.16-Figure C.17 Power Transformation Model.

Figure C.18-Figure C.21 Individual fitted Models.

Figure C.22 Group shelf-life estimation.

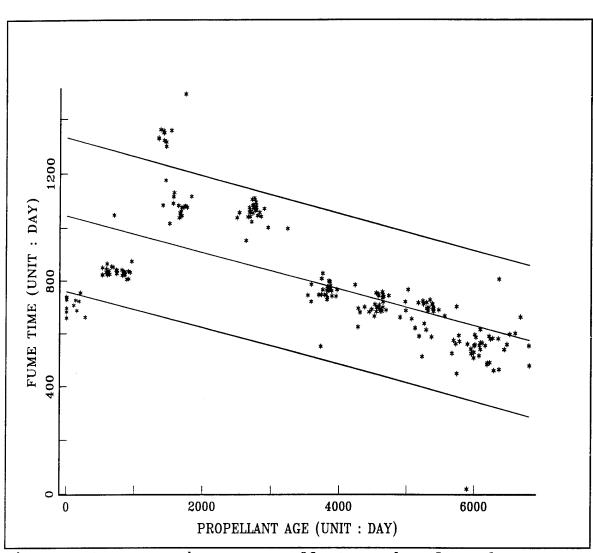


Figure C.1. Fume time vs propellant age based on the standard linear regression model I.

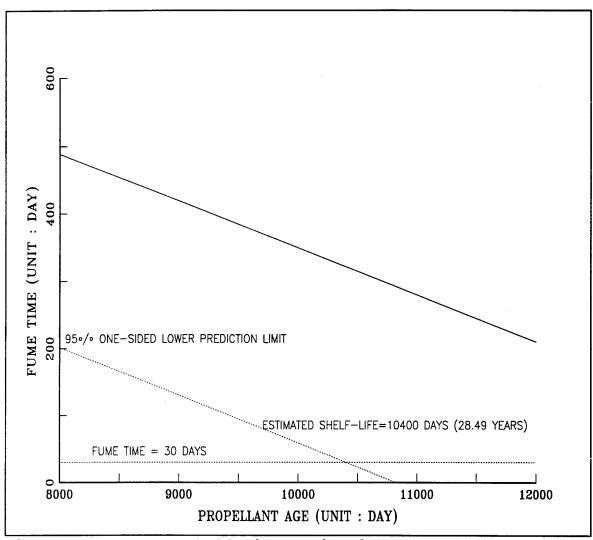


Figure C.2. Group shelf-life estimation based on the standard linear regression model I.

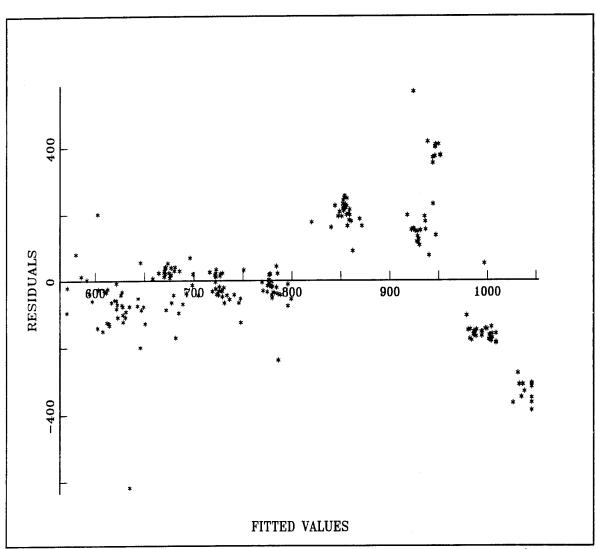


Figure C.3. Residual analysis based on the standard linear regression model I.

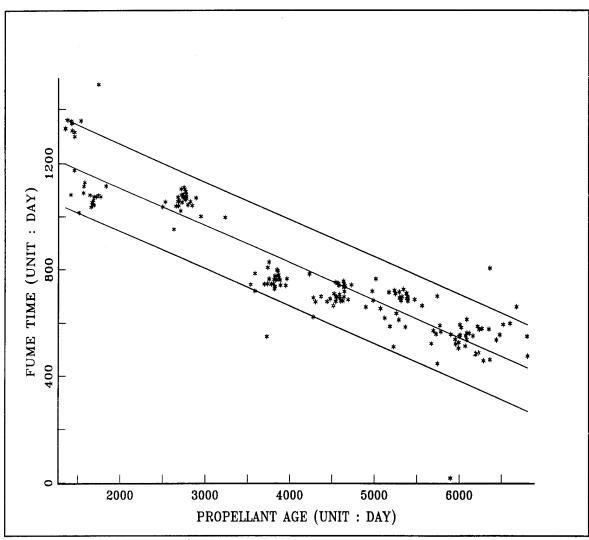


Figure C.4. Fume time vs propellant age based on the standard linear regression model II.

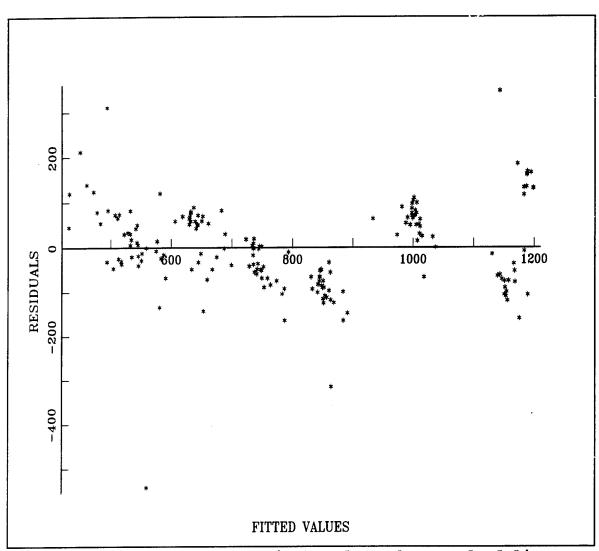


Figure C.5. Residual analysis based on the standard linear regression model II.

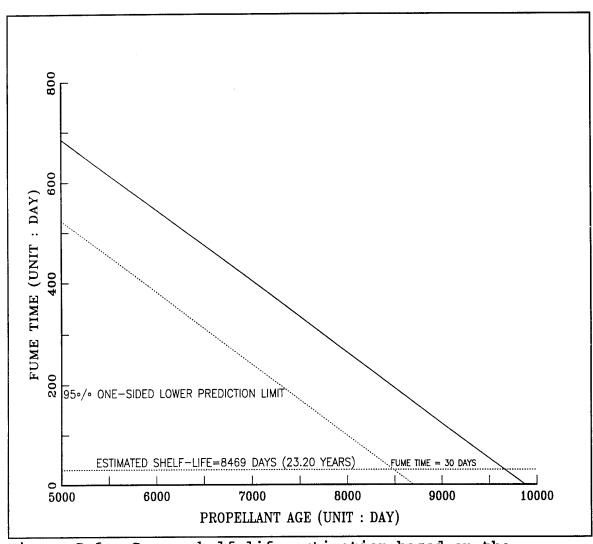


Figure C.6. Group shelf-life estimation based on the standard linear regression model II.

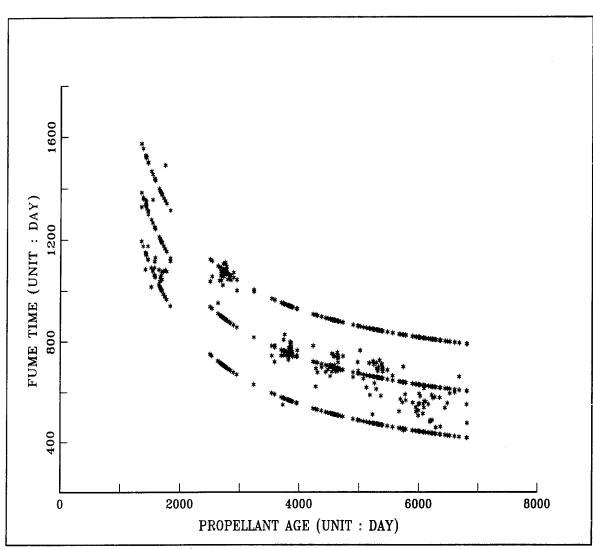


Figure C.7. Fume time vs propellant age based on the linearizing transformation model I.

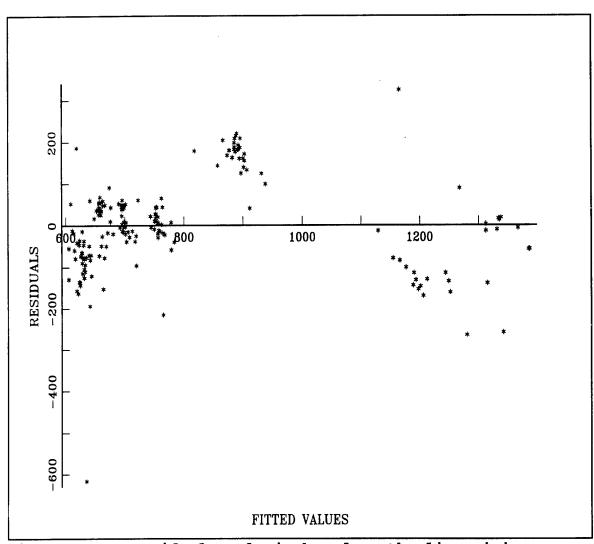


Figure C.8. Residual analysis based on the linearizing transformation model I.

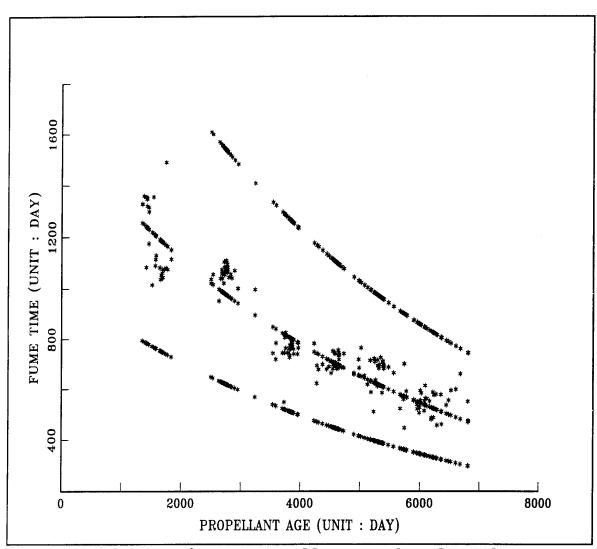


Figure C.9. Fume time vs propellant age based on the linearizing transformation model II.

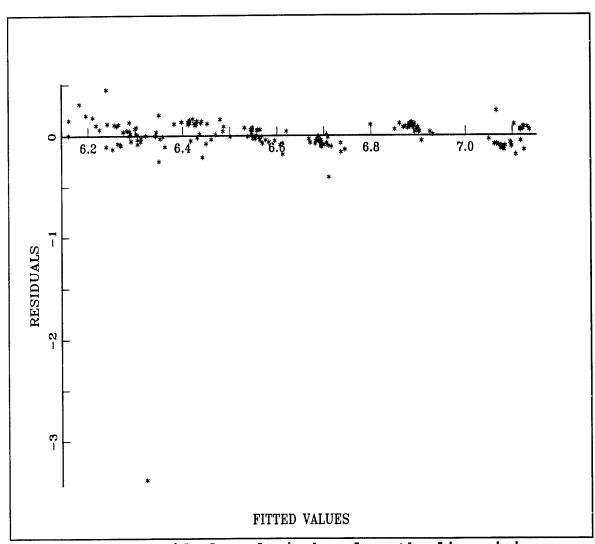


Figure C.10. Residual analysis based on the linearizing transformation model II.

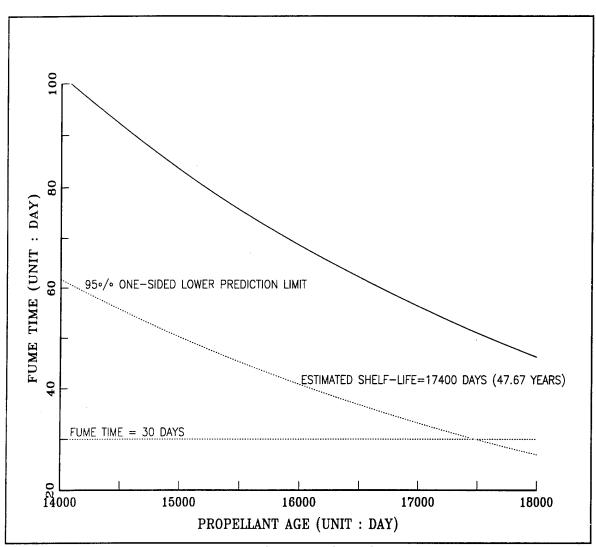


Figure C.11. Group shelf-life estimation based on the linearizing transformation model II.

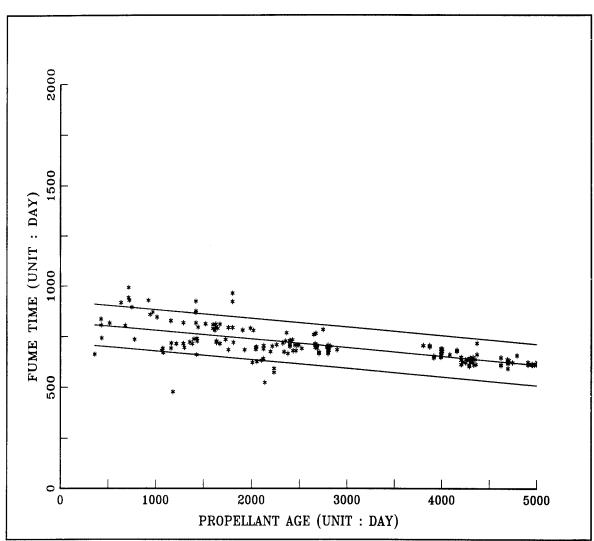


Figure C.12. Fume time vs propellant age based on the fleet return data.

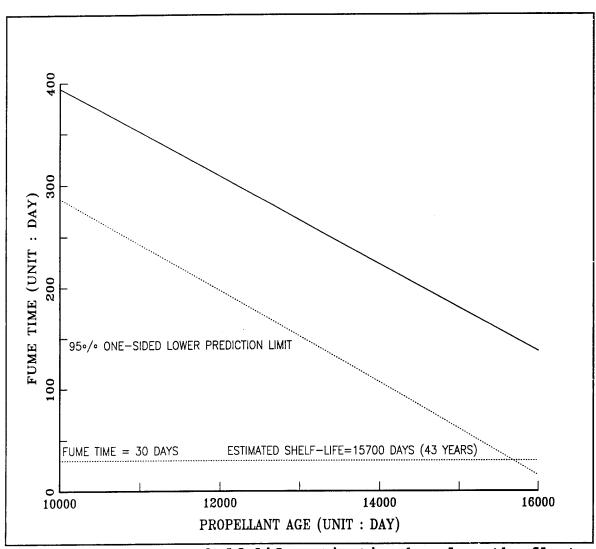


Figure C.13. Group shelf-life estimation based on the fleet return data

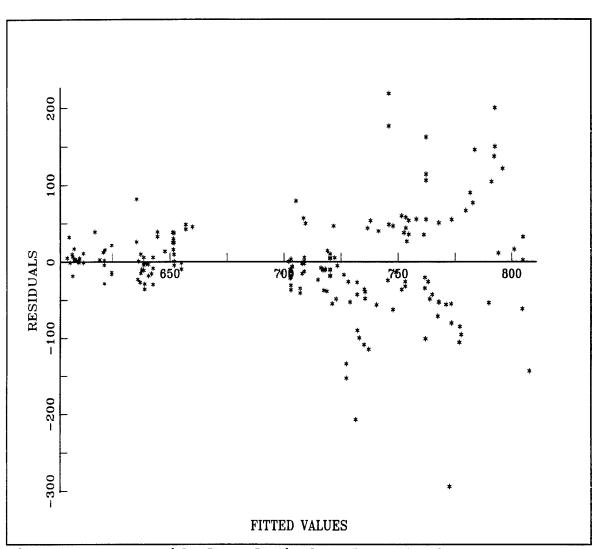


Figure C.14. Residual analysis based on the fleet return data

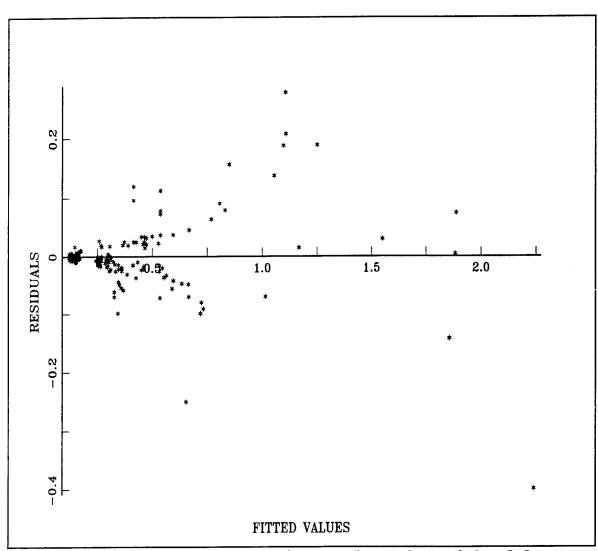


Figure C.15. Residual analysis based on the weighted least square model.

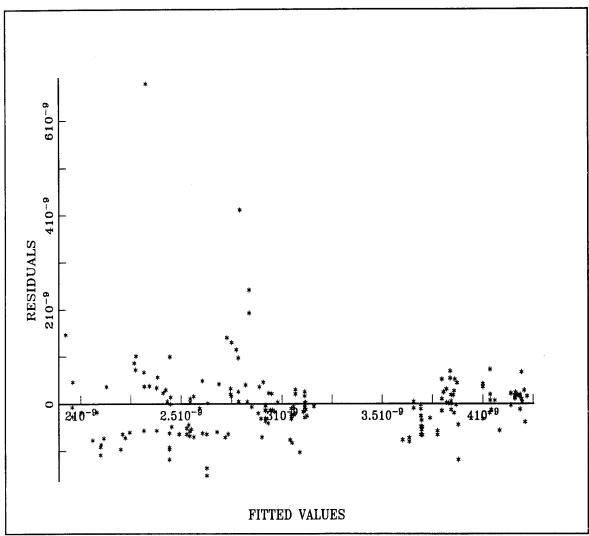


Figure C.16. Residual analysis based on the power transformation model.

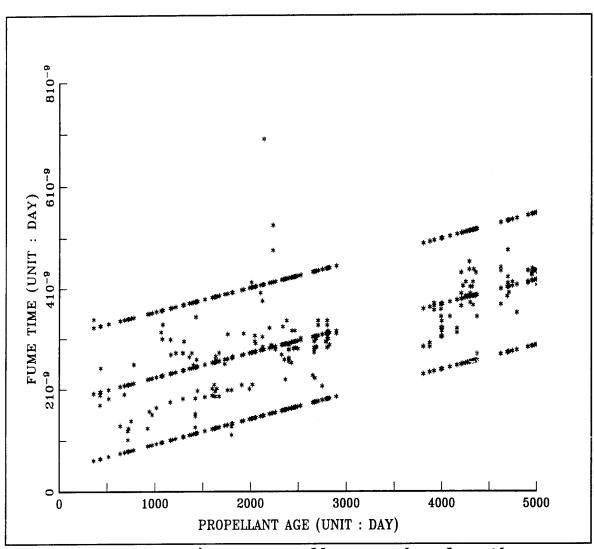


Figure C.17. Fume time vs propellant age based on the power transformation model.

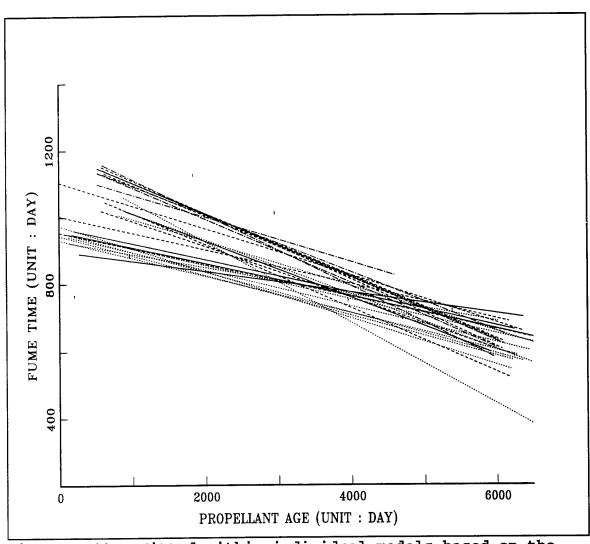


Figure C.18. Fitted within-individual models based on the standard linear regression model I.

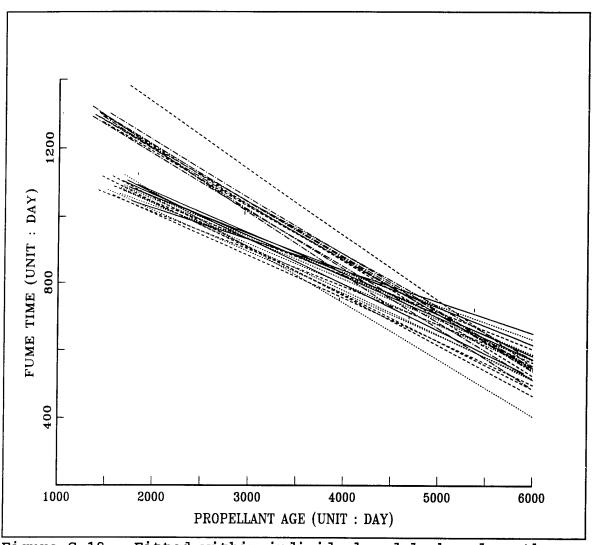


Figure C.19. Fitted within-individual models based on the standard linear regression model II.

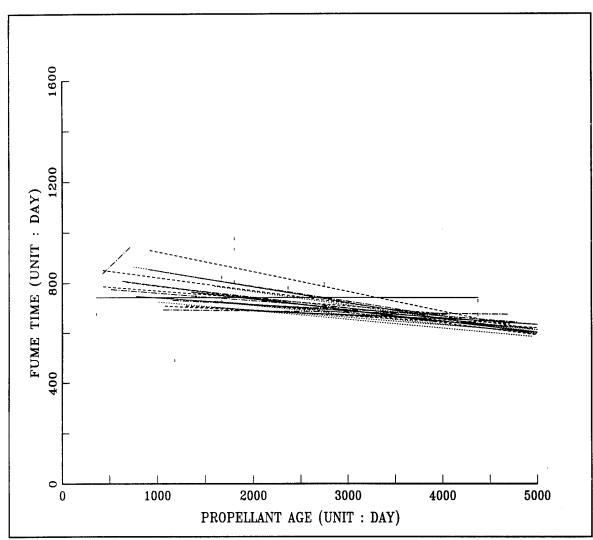


Figure C.20. Fitted within-individual models based on the fleet return data.

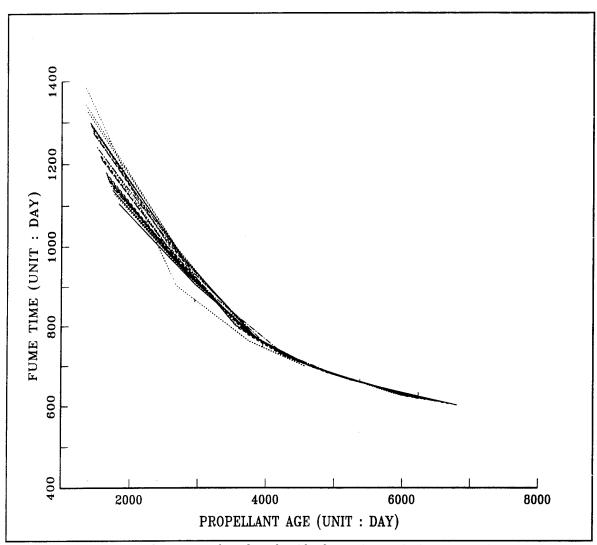


Figure C.21. Fitted within-individual models based on the linearizing transformation model I.

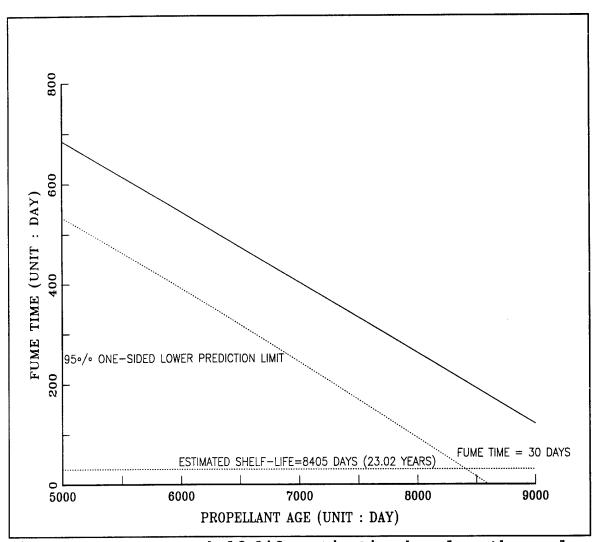


Figure C.22. Group shelf-life estimation based on the random effects regression model.

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